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September 29, 2004 *CTC*/DP-CL4528-04

U.S. Army Environmental Center
ATTN: SFIM-AEC-ATT (Mr. Richard Eichholtz)
5179 Hoadley Road
Aberdeen Proving Ground, MD 21010-5401

SUBJECT: Final Joint Test Protocol JP-P-1-1 for Validation of Alternatives to Lead-Containing Dry Film Lubricants for Antigalling/Antifretting, Antiseizing, and Assembly Aid Applications (CDRL A005), dated September 29, 2004

REFERENCE:
(1) Electronic Mail to Darnella Parker (*CTC*) from Darlene Bader-Lohn (USAEC), Approval dated September 28, 2004, Subject: NDCEE Task 272 Joint Test Protocol (JP-P-1-1) for Validation of Alternatives to Lead-Containing Dry Film Lubricants for Antigalling/Antifretting, Antiseizing, and Assembly Aid Applications, revised date July 28, 2004
(2) Task No 272, "Joint Group on Pollution Prevention (JG-PP) Program," approved December 18, 1999
(3) Contract Number DAAE30-98-C-1050

Dear Mr. Eichholtz:

Concurrent Technologies Corporation (*CTC*) is pleased to submit one (1) copy of the Subject Deliverable in response to Reference (1) Government approval and in accordance with the Reference (2) Task under the Reference (3) Contract. If you should require technical clarification, please call Mr. John Thomstatter, at (814) 269-2508. For contractual issues, please call the undersigned at the above direct dial number.

Very truly yours,

// Original Singed //

Darnella Parker
Manager, Contract Resources

/bem

Enclosures: as stated

cc: Ms. C. Brown, NASA AP2 Program Manager
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Engineering and Technical Services for Joint Group on Acquisition Pollution Prevention (JG-PP)

Final Joint Test Protocol JP-P-1-1

for Validation of Alternatives to Lead-Containing Dry Film Lubricants for Antigalling/Antifretting, Antiseizing, and Assembly Aid Applications

September 29, 2004

Contract No. DAAE30-98-C-1050
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Prepared by:
National Defense Center for Environmental Excellence

Operated by: Concurrent Technologies Corporation
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This revision to the Joint Test Report for Validation of Alternatives to Lead-Containing Dry Film Lubricants for Antigalling/Antifretting, Antiseizing, and Assembly Aid Applications includes an additional test requirement for humidity resistance. This requirement was identified by turbine engine original equipment manufacturers based on experience in evaluating water-based dry film lubricants (DFLs) for antigalling/antifretting applications. Exposure of some DFLs to hot, humid conditions has the potential to rehydrate the binder, rendering the DFL as a “wet” coating that is susceptible to removal.

This report was prepared by the NDCEE. This report was prepared on behalf of, and under guidance provided by the Propulsion Environmental Working Group (PEWG) and the Joint Group on Pollution Prevention (JG-PP). The structure, format, and depth of technical content of the report were determined by the JG-PP Working Group, PEWG, original equipment manufacturers, and other Government technical representatives in response to the specific needs of this project.

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LIST OF ACRONYMS

AISI	American Iron and Steel Institute
AMS	Aerospace Materials Specifications
AS	Aerospace Standard
ASTM	American Society for Testing and Materials
Cr	chromium
CTC	Concurrent Technologies Corporation
CTG	Control Technique Guidelines
DCMA	Defense Contract Management Agency
DFL	dry film lubricant
DLA	Defense Logistics Agency
DoD	Department of Defense
DTA	Differential Thermal Analysis
EPA	Environmental Protection Agency
GEAE	General Electric Aircraft Engines
JG-PP	Joint Group on Pollution Prevention
JLC	Joint Logistics Commanders
JTP	Joint Test Protocol
JTR	Joint Test Report
KSI	kilo PSI
LVDT	linear variable differential transducer
Na_2SO_4	sodium sulfate
NaCl	sodium chloride
NASA	National Aeronautics and Space Administration
NDCEE	National Defense Center for Environmental Excellence
NESHAP	National Emission Standards for Hazardous Air Pollutants
OEM	original equipment manufacturer
P&W-UTC	Pratt and Whitney, United Technologies Corporation
PEWG	Propulsion Environmental Working Group
PTFE	polytetrafluoroethylene
RACT	Reasonable Available Control Technology
SAE	Society of Automotive Engineers
SCAQMD	Southern Coast Air Quality Management District
TGA	Thermalgravimetric Analysis
TR	technical representative
VOC	volatile organic compound
MEK	methyl ethyl ketone

FOREWORD

This revision to the Joint Test Report for Validation of Alternatives to Lead-Containing Dry Film Lubricants for Antigalling/Antifretting, Antiseizing, and Assembly Aid Applications includes an additional test requirement for humidity resistance (Section 3.18). This requirement was identified by turbine engine original equipment manufacturers based on experience in evaluating water-based dry film lubricants (DFLs) for antigalling/antifretting applications. Exposure of some DFLs to hot, humid conditions has the potential to rehydrate the binder, rendering the DFL as a “wet” coating that is susceptible to removal.

Minor changes were also made in the introduction section to update Joint Group on Pollution Prevention terminology.

PREFACE

This report was prepared by the National Defense Center for Environmental Excellence (NDCEE), operated by Concurrent Technologies Corporation (*CTC*). This report was prepared on behalf of, and under guidance provided by the Propulsion Environmental Working Group (PEWG) and the Joint Group on Pollution Prevention (JG-PP). The structure, format, and depth of technical content of the report were determined by the JG-PP Working Group, PEWG, original equipment manufacturers, and other Government technical representatives in response to the specific needs of this project.

We wish to acknowledge the invaluable contributions provided by the following organizations involved in the creation of this document:

Aeronautical System Center, Environmental Management Office
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Naval Sea Systems Command
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Oklahoma City Air Logistics Center
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1. INTRODUCTION

This project is being conducted under the auspices of the Joint Group on Pollution Prevention (JG-PP) and the Propulsion Environmental Working Group (PEWG). The goal of this project is to eliminate lead as found in dry film lubricants (DFLs) used in aircraft engines.

Joint Group on Pollution Prevention (JG-PP): JG-PP is a partnership between the Military Services, National Aeronautics and Space Administration (NASA), Defense Logistics Agency (DLA), and Defense Contract Management Agency (DCMA), chartered by the Joint Logistics Commanders (JLC) to reduce or eliminate HazMats or processes within the acquisition and sustainment communities. By establishing these partnerships, JG-PP addresses the common problems through shared efforts to produce joint solutions.

The primary objectives of the JG-PP are to:

- identify shared opportunities
- facilitate partnerships
- facilitate qualification requirements
- reduce duplication of effort
- reduce risk
- reduce cost.

The Propulsion Environmental Working Group (PEWG): The PEWG is a tri-service forum established in 1991 by the Air Force Propulsion Product Group Manager and the Joint Propulsion Coordinating Committee to resolve common environmental issues and facilitate technical interchange between System Program Offices, Development System Offices, engine contractors, engine users/customers, the Air Force Propulsion Product Group Manager and tri-service team members. It serves as the hazardous material management subcommittee of the Joint Propulsion Coordinating Committee. Its goal is to integrate pollution prevention into the systems engineering process by facilitating the identification, tracking, elimination, substitution, and minimization of hazardous materials on all programs supported by team members. It is intended to assist Integrated Product Teams and engine programs in managing environmental and hazardous material related issues. The original equipment manufacturers (OEMs) involved with the PEWG project to eliminate the use of lead-containing dry film lubricants in engines are Allison Engine Company, Honeywell (formerly AlliedSignal), General Electric Aircraft Engines (GEAE), Pratt & Whitney-United Technologies Corporation (P&W-UTC), and Williams International.

This Joint Test Protocol (JTP) contains the tests necessary to qualify potential alternatives to the selected target HazMat and process, for particular applications. These tests were derived from engineering, performance, and operational impact (supportability) requirements defined by a consensus of government and industry participants. The requirements in this JTP were identified by multiple contractors for a

number of application categories. A candidate alternative may fail to meet one or more of these requirements but still be suitable for specific applications.

A subsequent Joint Test Report (JTR) will document the data and results of the testing. The JTR will then be made available as a reference for future pollution prevention efforts by other Department of Defense (DoD) and commercial users. Table 1 summarizes the target HazMat, process/material, application, current specifications, affected programs, and candidate parts/substrates.

Table 1. Target HazMat Summary

Target HazMat	Lead, as contained in dry film lubricants
Current Process/ Material	Dry Film Lubricants (Solid Film Lubricants)
Applications	Lubricants for aiding assembly and subsequent disassembly of mated parts (antiseizing) and/or for antigalling/antifretting
Current Specifications	MIL-F-7179, MIL-L-23398, MIL-L-45983, MIL-L-46010, MIL-L-46147, MIL-L-81329 AMS 2525, AMS 3084, AS 1701 A50TF9, A50TF79, A50TF147, A50TF150, A50TF159, A50TF170, A50TF171, A50TF174, A50TF192, A50TF279, EMS 5248, EMS 5402, EMS 27605, EMS 27608, EMS 27610, EMS 27615, EMS 27628, EPS 11705, EPS 11706, EPS 11708, EPS 11709, EPS 11710, EPS 11712, EPS 11715, EPS 11718, EPS 11720, F50TF42, F50TF58/70, F50TF88, GM6078M, PWA 586
Affected Programs*	**F100** in F-15 and F-16; **F101** in B-1B; **F103** in KC-10; **F107** in cruise missile; **F108** in KC-135R; **F110** in F-14 and F-16; **F112** in cruise missile; **F117** in C-17; **F118** in B-2 and U-2; **F119** in F-22; **F404/F414** in F/A-18 and F-117A; **J52** in A-4, A-6, EA-6; **T53** in UH-1; **T55** in CH-47 and MH-47; **T56** in C-130, E-2, P-3; **T64** in NH-53; **T406** in V-22 and C-130J; **T700** in UH-60 and AH-64; **T800** in Cheyenne; **TF30** in F-14 and EF-111; **TF33** in B-52, C-141, KC-135; **TF34** in A-10 and OA-10; **TF39** in C-5

(Table 1 continued on next page)

Table 1. Target HazMat Summary (continued)

Candidate Parts/ Substrates	Threaded fasteners, compressor and turbine discs and blade roots aluminum: 2024 cobalt: Haynes 188, MP159 magnesium: AMS 4375 nickel: Hastelloy X, AMS 5664, Inconel 718, Waspaloy steel: A-286, AISI 4340, Greek Ascoloy, AM-355, 17-4PH, AMS 5617, AISI 304, AISI 321, AISI 347 titanium: Ti-6Al-4V, Ti-8-1-1

* This table reflects families of engines; various models are included that are used on a number of platforms.

2. ENGINEERING, PERFORMANCE, AND TESTING REQUIREMENTS

A joint group led by JG-PP and PEWG and consisting of technical representatives from Allison Engine Company, AlliedSignal Engines, GEAE, P&W-UTC, Williams International, the affected DoD Program Managers, representatives of the Sustainment Community, and other government technical representatives identified engineering, performance, and operational impact (supportability) requirements. These requirements were identified for dry film lubricants for antiseizing, antigalling/antifretting, and assembly aid applications. This group then reached consensus on tests with procedures, methodologies, and acceptance criteria to qualify alternatives against these technical requirements. These tests were identified by multiple contractors for a number of application categories; failure in any test does not necessarily disqualify a candidate DFL for use in all possible applications.

Tests should be conducted in a manner that will eliminate duplication and maximize use of each test specimen. For example, where possible, more than one test should be performed on each specimen. The number and types of tests that can be run on any one specimen will be determined by the destructiveness of each test.

Tests in this JTP may involve the use of hazardous materials, operations, and equipment. This JTP does not address all of the safety issues associated with its use. It is the responsibility of each user of this JTP to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to its use.

There are a number of general application categories for DFLs defined in this JTP. These categories are as follows:

- **LG** - low temperature antigalling/antifretting applications (up to 850°F), DFL used to protect part surfaces against sliding and oscillating wear
- **HG** - high temperature antigalling/antifretting applications (850°F to 1400°F), DFL used to protect part surfaces against sliding and oscillating wear
- **LS** - low temperature antiseizing applications (up to 850°F), DFL applied to threaded fasteners at assembly to facilitate subsequent disassembly
- **HS** - high temperature antiseizing applications (850°F to 1400°F), DFL applied to threaded fasteners at assembly to facilitate subsequent disassembly
- **AD** - short term assembly aid applications, DFL used during assembly to prevent seizing and protect parts from nicks and scratches, DFLs for this application are usually applied by aerosol spray and are allowed to briefly air dry prior to assembly.

It is possible for a DFL to be a candidate for multiple applications (e.g., <u>both</u> LG and HG). In these cases, the DFL should be tested for all applications for which it is being considered. For example, where LG is indicated for testing, DFLs that are candidates for <u>only</u> LG applications or for <u>both</u> LG and HG applications should be tested. Similarly,

where HG is indicated for testing, DFLs that are candidates for only HG applications or for both LG and HG applications should be tested.

The individual OEMs participating in the PEWG may have different requirements for specific applications. For instance, while the high temperature application categories defined above extend up to 1400°F, it has been estimated that a dry film lubricant stable up to 1200°F is suitable for about 90 percent of high temperature antigalling/antifretting and antiseizing applications.

Each test described also specifies to which application category(ies) it applies. When candidate DFLs are selected for testing, they should also be associated with the application category(ies) for which they are best suited, and should only be subjected to the appropriate tests.

The engineering requirements for which the tests in this JTP were chosen are the following:

- Antiseizing - ability of cured DFL to reduce or prevent seizing of mated components
- Chemical Content - absence or acceptable concentrations of certain chemicals targeted for elimination or reduction to improve environmental or occupational health properties
- Chemical Resistance - ability of cured DFL to resist degradation or softening when placed in contact with selected chemicals
- Compatibility with Substrate - lack of degradation of substrate caused by contact with cured DFL
- Corrosion Protection - ability of cured DFL to not accelerate or to prevent corrosion of substrate when specimen is exposed to corrosive environment
- Film Properties - adhesion, thickness of cured film, uniformity of cured film, surface condition of applied film
- Lubricity (Coefficient of Friction) - ability of cured DFL to lubricate
- Thermal Stability (Useful Temperature Range) - ability of cured DFL to resist degradation within a given temperature range
- Wear Resistance - ability of cured DFL to withstand physical abrasion
- Humidity Resistance – ability of cured DFL to not re-hydrate in hot, humid conditions such that it can be wiped off of a substrate.

Table 2 lists all Engineering and Test Requirements identified by the JG-PP/PEWG participants for validating alternatives to lead-containing DFLs. This listing includes acceptance criteria and the references, if any, used for developing the tests. Table 3 lists the tests described in this JTP according to the application category. Note that not all tests listed in Table 3 are listed in Table 2; this is because some of the tests in Table 3 are suggested ongoing quality control tests rather than qualification tests. Quality control tests are described in Section 4.

Table 2. Engineering and Performance Test Requirements

Engineering Requirement	Test	JTP Section	Application Categories	Acceptance Criteria	References
Corrosion Protection	Aluminum Corrosion Resistance	3.1	AD	No discoloration, pitting, white deposits, or other evidence of corrosion greater than that observed on uncoated control specimens	ASTM D2649 - 83
Chemical Content	Chromium Content	3.2	LG, HG, LS, HS, AD	Chromium content below 100 ppm	ASTM D3718 - 85a
Film Properties	Cured Film Thickness Uniformity	3.3	LG, HG, LS, HS	No more than one thickness measurement per panel below 0.0003 inch (0.3 mil) and no more than one thickness measurement per panel above 0.0008 inch (0.8 mil)	ASTM E376 - 89 ASTM B244 - 79 ASTM D1400 - 87 ASTM B499 - 88 ASTM D1186 - 87
Film Properties (adhesion)	Dry Tape Adhesion	3.4	LG, HG, LS, HS, AD	No exposure of underlying substrate	ASTM D2510 - 83
Compatibility with Substrate, Thermal Stability	Elevated Temperature Material Compatibility	3.5	LG, HG, LS, HS	No substrate degradation exceeding by 0.0002 inches or more the degradation observed on the uncoated control specimens	*none*
Corrosion Protection	Fastener Corrosion	3.6	HS	No evidence of substrate corrosion greater than that of the uncoated control assemblies	*none*

(Table 2 continued on next page)

Table 2. Engineering and Performance Test Requirements (continued)

Engineering Requirement	Test	JTP Section	Application Categories	Acceptance Criteria	References
Chemical Resistance, Film Properties (adhesion)	Fluid Resistance	3.7	LG, HG, LS, HS	No lifting, softening, blistering, cracking, peeling, significant discoloration, or loss of adhesion	ASTM D2510 - 83 ASTM D1141 - 90 ASTM D1193 - 91 MIL-A-8243D MIL-H-87257 MIL-L-23699E MIL-T-5624R MIL-T-83133D VV-D-1078B
Chemical Content	Lead and Cadmium Content	3.8	LG, HG, LS, HS, AD	No more than 100 ppm lead or cadmium	ASTM D3335 - 85a
Wear Resistance, Lubricity	Reciprocating Sliding Wear	3.9	LG, HG	Residual film of lubricant with smooth or slightly striated wear pattern remaining on shoe specimen; no DFL flaking, base metal wear, or other signs of degradation Coefficient of Kinetic Friction: LG: less than 0.12 HG: less than 0.15	ASTM G115 - 93
Corrosion Protection	Salt Spray (Fog) Corrosion Resistance	3.10	LG	No more than three (3) corrosion spots per specimen and no corrosion spots larger than 1.0 millimeter diameter	ASTM B117 - 94 ASTM D165 - 92
Chemical Resistance, Film Properties (adhesion)	Solvent Rub	3.11	LG, HG, LS, HS	No separation of lubricant film or exposure of substrate	*none*

(Table 2 continued on next page)

Table 2. Engineering and Performance Test Requirements (continued)

Engineering Requirement	Test	JTP Section	Application Categories	Acceptance Criteria	References
Corrosion Protection	Stress Corrosion	3.12	LG, LS, AD	No cracking of substrate	ASTM F945 - 85
Corrosion Protection	Sulfurous Acid Salt Spray	3.13	LG, AD	No pitting, staining, or other visible evidence of corrosion	ASTM B117 – 94 ASTM D1141 – 90
Thermal Stability, Film Properties (adhesion)	Thermal Shock Stability	3.14	LG, HG, LS, HS	No flaking, cracking, softening, lifting, or loss of adhesion greater than that observed for the control (lead containing DFL)	ASTM D2511 - 83 ASTM D2510 - 83
Thermal Stability	Thermal Stability by Simultaneous Differential Thermal Analysis-Thermogravimetric Analysis (SDT)	3.15	LG, HG, LS, HS	This test is intended to provide baseline information on the temperature limits of a DFL. The temperature limits will define and categorize the DFL as low temperature (up to 850°F) or high temperature (between 850°F and 1400°F). The *useful temperature limit* will be defined as the temperature above 400°F at which there is a substantial change (increase or decrease) in the mass of the cured DFL sample.	*none*

(Table 2 continued on next page)

Table 2. Engineering and Performance Test Requirements (continued)

Engineering Requirement	Test	JTP Section	Application Categories	Acceptance Criteria	References
Antiseizing, Thermal Stability	Torque-Tension Evaluation	3.16	LS, HS	*See test description, starting on Page 53*	*none*
Chemical Content	Volatile Organic Compound Content	3.17	LG, HG, LS, HS, AD	VOC content no greater than 500 g/L for DFLs supplied as bulk liquid and VOC content no greater than 880 g/L for DFLs supplied in aerosol cans	ASTM D1475 - 90 ASTM D2369 - 92 ASTM D3792 - 91 ASTM D3960 - 92 ASTM D4017 - 90 ASTM D4457 – 85
Humidity Resistance	Humidity Resistance	3.18	LG, HG	Minimal or no removal of DFL coating observed over the length of the testing	*none*

Table 3. Tests for each Application Category

Application Category	Test Name	JTP Section	Qualification Test	Quality Control Test
LG low temperature antigalling/ antifretting	Chromium Content	3.2	Y	
	Cured Film Thickness Uniformity	3.3	Y	
	Dry Tape Adhesion	3.4	Y	
	Elevated Temperature Material Compatibility	3.5	Y	
	Fluid Resistance	3.7	Y	
	Lead and Cadmium Content	3.8	Y	
	Reciprocating Sliding Wear	3.9	Y	
	Salt Spray (Fog) Corrosion Resistance	3.10	Y	
	Solvent Rub	3.11	Y	
	Stress Corrosion	3.12	Y	
	Sulfurous Acid Salt Spray	3.13	Y	
	Thermal Shock Stability	3.14	Y	
	Thermal Stability by Simultaneous Differential Thermal Analysis - Thermogravimetric Analysis (SDT)	3.15	Y	
	Volatile Organic Compound Content	3.17	Y	
	Humidity Resistance	3.18	Y	
	Endurance Life	4.1		Y
	Load Carrying Capacity	4.2		Y

(Table 3 continued on next page)

Table 3. Tests for each Application Category (continued)

Application Category	Test Name	JTP Section	Qualification Test	Quality Control Test
HG high temperature antigalling/ antifretting	Chromium Content	3.2	Y	
	Cured Film Thickness Uniformity	3.3	Y	
	Dry Tape Adhesion	3.4	Y	
	Elevated Temperature Material Compatibility	3.5	Y	
	Fluid Resistance	3.7	Y	
	Lead and Cadmium Content	3.8	Y	
	Reciprocating Sliding Wear	3.9	Y	
	Solvent Rub	3.11	Y	
	Thermal Shock Stability	3.14	Y	
	Thermal Stability by Simultaneous Differential Thermal Analysis - Thermogravimetric Analysis (SDT)	3.15	Y	
	Volatile Organic Compound Content	3.17	Y	
	Humidity Resistance	3.18	Y	
	Endurance Life	4.1		Y
	Load Carrying Capacity	4.2		Y

(Table 3 continued on next page)

Table 3. Tests for each Application Category (continued)

Application Category	Test Name	JTP Section	Qualification Test	Quality Control Test
LS low temperature antiseizing	Chromium Content	3.2	Y	
	Cured Film Thickness Uniformity	3.3	Y	
	Dry Tape Adhesion	3.4	Y	
	Elevated Temperature Material Compatibility	3.5	Y	
	Fluid Resistance	3.7	Y	
	Lead and Cadmium Content	3.8	Y	
	Solvent Rub	3.11	Y	
	Stress Corrosion	3.12	Y	
	Thermal Shock Stability	3.14	Y	
	Thermal Stability by Simultaneous Differential Thermal Analysis - Thermogravimetric Analysis (SDT)	3.15	Y	
	Torque-Tension Evaluation	3.16	Y	
	Volatile Organic Compound Content	3.17	Y	

(Table 3 continued on next page)

Table 3. Tests for each Application Category (continued)

Application Category	Test Name	JTP Section	Qualification Test	Quality Control Test
HS high temperature antiseizing	Chromium Content	3.2	Y	
	Cured Film Thickness Uniformity	3.3	Y	
	Dry Tape Adhesion	3.4	Y	
	Elevated Temperature Material Compatibility	3.5	Y	
	Fastener Corrosion	3.6	Y	
	Fluid Resistance	3.7	Y	
	Lead and Cadmium Content	3.8	Y	
	Solvent Rub	3.11	Y	
	Thermal Shock Stability	3.14	Y	
	Thermal Stability by Simultaneous Differential Thermal Analysis - Thermogravimetric Analysis (SDT)	3.15	Y	
	Torque-Tension Evaluation	3.16	Y	
	Volatile Organic Compound Content	3.17	Y	
AD assembly aid (air dry)	Aluminum Corrosion Resistance	3.1	Y	
	Chromium Content	3.2	Y	
	Dry Tape Adhesion	3.4	Y	
	Lead and Cadmium Content	3.8	Y	
	Stress Corrosion	3.12	Y	
	Sulfurous Acid Salt Spray	3.13	Y	
	Volatile Organic Compound Content	3.17	Y	

3. TEST DESCRIPTIONS

Tests identified in Table 2 are further defined below to include test description, rationale, and methodology. Also included as needed are any major or unique equipment and instrumentation, and data analysis procedures. Test methodology includes the definition of test parameters and conditions, test specimens/substrates, experimental control specimens, and acceptance criteria.

The number and type of test specimens prescribed in the test methodology is the type and number per individual candidate DFL. Each test may be performed using a currently accepted lead-containing DFL, uncoated substrate specimen(s), or both as experimental controls; the test methodology specifies what experimental control specimens, if any, are required for each test.

For each test, the candidate alternative DFL(s) should be applied to the test specimens and cured in accordance with the manufacturer's recommendations. After application, the DFL(s) must be visually examined under normal work lighting conditions and 3X magnification, and used for testing only if the DFL has formed a smooth film of uniform color with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping. If any film defects are noted, the specimen must be excluded from testing; it may be possible to remove the DFL film and reapply an acceptable film in order to make use of the substrate.

The film thickness of the DFL(s) for the tests described in Sections 3.1, 3.3 through 3.5, 3.7, and 3.10 through 3.14 should be 0.3 to 0.8 mils. This film thickness was chosen by the participants as representative of the most common film thickness for currently used DFLs. These film thicknesses must be verified by measurement at three (3) separate locations on each test specimen; except for the Cured Film Thickness Uniformity Test (Section 3.3), which requires ten (10) separate measurements. These thickness measurements must be recorded and reported with the test results.

All LG candidates for the Reciprocating Sliding Wear test (Section 3.9) must be applied to a thickness of 0.9 to 1.1 mil, while HG candidates for the Reciprocating Sliding Wear test must be applied to a thickness of 0.7 to 0.9 for fretting tests and 0.2 to 1.0 mil for galling tests. The film thicknesses for the Reciprocating Sliding Wear test were chosen based on current practice in testing DFLs intended to prevent galling and fretting. The film thickness required for testing of HG candidates is smaller than the film thickness for LG candidates because currently used lead containing HG category DFLs are generally not applied in as thick a film as LG category DFLs. These film thicknesses must be verified by at least one measurement on each specimen. Multiple film thickness measurements for each specimen are preferred, but the small size of the specimens used for the Reciprocating Sliding Wear test may preclude multiple independent film thickness measurements. These thickness measurements must be recorded and reported with the test results.

The test descriptions in this JTP apply to DFLs for a number of applications and

operating temperature regimes. These categories are antigalling/antifretting, low temperature applications (LG); antigalling/antifretting, high temperature applications (HG); antiseizing low temperature applications (LS); antiseizing high temperature applications (HS); and air-dry assembly aid applications (AD). Please note that the tests described below are intended to validate performance of candidate DFLs for the broad application categories defined above. Failure in any individual test does not necessarily disqualify a candidate DFL for use in a specific application.

Below is a listing of substrate types that should be used for testing:

Table 4. Test Specimen Codes and Substrate Descriptions

Test Specimen Code	Substrate Description
AL1a	Aluminum alloy 2024-T3, bare, conforming to ASTM B209 - 92a and AMS 4037M; chromic acid anodized in accordance with ASTM D1730 - 67, Type C, Method 2; 3" x 6" x 0.020" (minimum) thick panels; for Aluminum Corrosion Resistance test (Section 3.1), Dry Tape Adhesion Test (Section 3.4), Fluid Resistance test (Section 3.7), and Solvent Rub test (Section 3.11)
AL1b	Aluminum alloy 2024-T3, bare, conforming to ASTM B209 - 92a and AMS 4037M; sulfuric acid anodized in accordance with ASTM D1730 - 67, Type C, Method 1; 3" x 6" x 0.020" (minimum) thick panels; for Aluminum Corrosion Resistance test (Section 3.1)
AL1c	Aluminum alloy 2024-T3, bare, conforming to AMS 4037M (4.4Cu 1.5Mg 0.60Mn, solution heat treated), not anodized • 3" x 6" x 0.050" (minimum) thick panels, for Cured Film Thickness Uniformity test (Section 3.3) • 1" x 0.5" x 0.050" (minimum) thick specimens, for Elevated Temperature Material Compatibility test (Section 3.5)
CO1	Corrosion and heat resistant cobalt alloy Haynes 188 conforming to AMS 5608C (40Co 22Cr 22Ni 14.5W 0.07La, solution heat treated), 1" x 0.5" x 0.062" (minimum) thick specimens, for Elevated Temperature Material Compatibility test (Section 3.5)

(Table 4 continued on next page)

Table 4. Test Specimen Codes and Substrate Descriptions (continued)

Test Specimen Code	Substrate Description
CO2	Corrosion and heat resistant cobalt alloy MP159, solution heat treated • 1" x 0.5" x 0.050" (minimum) thick specimens conforming to AMS 5843C, for Elevated Temperature Material Compatibility test (Section 3.5) • Bolts conforming to AS 7475, for Fastener Corrosion test (Section 3.6), of the following diameters: ➢ 0.375 inch ➢ 0.500 inch
MG	Magnesium alloy conforming to AMS 4375J (3.0Al 1.0Zn 0.20Mn, annealed and recrystallized), 1" x 0.5" x 0.032" (minimum) thick specimens, for Elevated Temperature Material Compatibility test (Section 3.5)
NI1	Corrosion and heat resistant nickel alloy Hastelloy X conforming to AMS 5536L (47.5Ni 22Cr 1.5Co 9.0Mo 0.60W 18.5Fe, solution heat treated), 1" x 0.5" x 0.035" (minimum) thick specimens, for Elevated Temperature Material Compatibility test (Section 3.5)

(Table 4 continued on next page)

Table 4. Test Specimen Codes and Substrate Descriptions (continued)

Test Specimen Code	Substrate Description
NI2a	Corrosion and heat resistant nickel alloy Waspaloy; solution, stabilization, and precipitation heat treated • 1" x 0.5" x 0.050" (minimum) thick specimens conforming to AMS 5709F, for Elevated Temperature Material Compatibility test (Section 3.5) • 3" x 6" x 0.050" (minimum) thick panels conforming to AMS 5709F, for Thermal Shock Stability test (Section 3.14) • Self-locking nuts conforming to AS 7253, for Fastener Corrosion test (Section 3.6) and Torque-Tension Evaluation (Section 3.16), of the following diameters: ➢ 0.250 inch ➢ 0.500 inch • Bolts conforming to AS 7471, for Fastener Corrosion test (Section 3.6) and Torque-Tension Evaluation (Section 3.16), of the following diameters: ➢ 0.250 inch ➢ 0.500 inch • Blocks conforming to AMS 5709F, of dimensions suitable for Torque-Tension Evaluation (Section 3.16)
NI2b	Corrosion and heat resistant nickel alloy Waspaloy conforming to AMS 5544G (57Ni 19.5Cr 13.5Co 4.2Mo 3.0Ti 1.4Al 0.05Zr 0.006B, consumable electrode vacuum induction melted, annealed), 1" x 0.5" x 0.050" (minimum) thick specimens, for Elevated Temperature Material Compatibility test (Section 3.5)

(Table 4 continued on next page)

Table 4. Test Specimen Codes and Substrate Descriptions (continued)

Test Specimen Code	Substrate Description
NI3	Corrosion and heat resistant nickel alloy Inconel 718, 1775°F solution heat treated • Self-locking nuts conforming to AMS 5662J, for Fastener Corrosion test (Section 3.6) and Torque-Tension Evaluation (Section 3.16), of the following diameters: ➢ 0.250 inch ➢ 0.500 inch • Bolts conforming to AS 7467, for Fastener Corrosion test (Section 3.6) and Torque-Tension Evaluation (Section 3.16), of the following diameters: ➢ 0.250 inch ➢ 0.500 inch • Shoe and block specimens conforming to AMS 5662J, for Reciprocating Sliding Wear Test (Section 3.9) • Blocks conforming to AMS 5662J, of dimensions suitable for Torque-Tension Evaluation (Section 3.16) • 3” x 6” x 0.020” thick test panels, for Humidity Resistance (Section 3.18)
ST1	Cold finished carbon steel AISI 1010 conforming to ASTM A108 - 90a, phosphate coated in accordance with AMS 2481F • 3” x 6” x 0.063” (minimum) thick panels, for Salt Spray (Fog) Corrosion Resistance test (Section 3.10)
ST2a	Corrosion resistant steel AISI 321, conforming to ASTM A167 - 92b, annealed, phosphate coated in accordance with AMS 2481F, 3” x 6” x 0.050” (minimum) thick panels, for Fluid Resistance test (Section 3.7)
ST2b	Corrosion resistant steel AISI 321, conforming to ASTM A167 - 92b; annealed; passivated in low temperature nitric acid solution in accordance with QQ-P-35C, Type VI; 3” x 6” x 0.050” (minimum) thick panels, for Cured Film Thickness Uniformity test (Section 3.3), Dry Tape Adhesion Test (Section 3.4), and Solvent Rub test (Section 3.11)

(Table 4 continued on next page)

Table 4. Test Specimen Codes and Substrate Descriptions (continued)

Test Specimen Code	Substrate Description
ST3	Low alloy steel AISI 4340 conforming to AMS 6359F (0.80Cr 1.8Ni 0.25Mo 0.38-0.43C), 1" x 0.5" x 0.550" (minimum) thick specimens, for Elevated Temperature Material Compatibility test (Section 3.5)
ST4	Corrosion and heat resistant steel alloy Greek Ascoloy conforming to AMS 5508E (13Cr 2.0Ni 3.0W, annealed), 1" x 0.5" x 0.050" (minimum) thick specimens, for Elevated Temperature Material Compatibility test (Section 3.5)
ST5	Corrosion resistant steel alloy 17-4PH, solution heat treated, precipitation hardenable • 3" x 6" x 0.050" (minimum) thick panels conforming to AMS 5604D, for Thermal Shock Stability test (Section 3.14)
ST9a	Corrosion and heat resistant precipitation hardenable iron alloy A-286 conforming to AMS 5858B, 1800°F solution heat treated, 1" x 0.5" x 0.050" (minimum) thick panels, for Elevated Temperature Material Compatibility test (Section 3.5)
ST9b	Corrosion and heat resistant precipitation hardenable iron alloy A-286, solution and precipitation heat treated • Self-locking nuts conforming to AS 7250, for Fastener Corrosion test (Section 3.6) and Torque-Tension Evaluation (Section 3.16), of the following diameters: ➢ 0.250 inch ➢ 0.500 inch • Bolts conforming to AS 7477A, for Fastener Corrosion test (Section 3.6), of the following diameters: ➢ 0.190 inch ➢ 0.375 inch ➢ 0.500 inch

(Table 4 continued on next page)

Table 4. Test Specimen Codes and Substrate Descriptions (continued)

Test Specimen Code	Substrate Description
ST10	Corrosion and heat resistant steel alloy AM-355 • Shoe and block specimens conforming to AMS 5547F for Reciprocating Sliding Wear test (Section 3.9) • 3" x 6" panels for Sulfurous Acid Salt Spray test (Section 3.13)
TI1a	Titanium alloy Ti-6Al-4V, annealed • Shoe and block specimens conforming to AMS 4967G (heat treatable), for Reciprocating Sliding Wear test (Section 3.9) • 5.6” x 0.5” x 0.050” thick specimens conforming to AMS 4911H, for Stress Corrosion test (Section 3.12) • Bolts conforming to AS 7460, for Torque-Tension Evaluation (Section 3.16), of the following diameters: ➢ 0.250 inch ➢ 0.500 inch • Blocks conforming to AMS 4967G, of dimensions suitable for Torque-Tension Evaluation (Section 3.16) • 3” x 6” x 0.020” thick test panels, for Humidity Resistance (Section 3.18)
TI1b	Titanium alloy Ti-6Al-4V, annealed, shot peened with CS110 steel shot to intensity level of 3 to 5A in accordance with AMS 2430L • Shoe and block specimens conforming to AMS 4967G, for Reciprocating Sliding Wear test (Section 3.9) • 3” x 6” x 0.050” (minimum) thick panels, conforming to AMS 4911H, for Thermal Shock Stability test (Section 3.14)
TI2	Titanium alloy Ti-8-1-1 conforming to AMS 4916F (8Al 1Mo 1V, duplex annealed), 5.6” x 0.5” x 0.050” specimens, for Stress Corrosion test (Section 3.12)

3.1. Aluminum Corrosion Resistance

Application Categories - AD

Test Description

This test allows evaluation of the corrosion characteristics of aluminum alloy 2024 coated with the DFL and exposed to high humidity, to demonstrate whether or not the DFL accelerates corrosion.

Apply DFL to 3" x 6" x 0.020" thick test panels as recommended by the DFL manufacturer, to a thickness of 0.3 to 0.8 mils, and cure. Verify the film thickness by measurement at three separate locations on each panel. Visually examine the panels under normal work lighting and 3X magnification, to verify that the applied DFL is a smooth film of uniform color, with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping.

Evaluate the corrosion resistance of aluminum coated with each candidate DFL in accordance with ASTM D2649 - 83 (*Standard Test Method for Corrosion Characteristics of Solid Film Lubricants*, approved March 25, 1983).

> Method Synopsis: Place a coated test panel and an uncoated panel of the same size and material together between an aluminum channel, in accordance with ASTM D2649 - 83. Make three (3) assemblies for each candidate DFL. In addition, make one (1) assembly with two (2) uncoated panels, as an experimental control. Place the assemblies in an oven for two hours as specified in Test Methodology. Remove the assemblies from the oven, and place in a humidity cabinet as specified in Test Methodology and in accordance with ASTM D2649 - 83. Remove the assemblies from the humidity cabinet after 500 hours of exposure, disassemble, and visually examine the test panels.

Rationale

This test of corrosion susceptibility of aluminum coated with the candidate DFL is similar to the requirements specified in MIL-L-46010E (*Lubricant, Solid Film, Heat Cured, Corrosion Inhibiting;* issued April 11, 1997), with the exception that the acceptance criteria used in MIL-L-46010E are not based on a comparison between DFL coated and uncoated specimens. The participants in the JG-PP/PEWG effort agreed that this test is necessary to determine whether or not the use of a candidate DFL as a short-term assembly aid will increase the susceptibility of aluminum parts to corrosion.

Test Methodology

Parameters	• 150 ± 8°F (65.5 ± 4°C) for 2 hours followed by • 120 ± 5°F (49 ± 3°C) at 95 ± 3% relative humidity for 500 hours
Number and Type of Specimens per Candidate DFL	Each specimen is 1 DFL coated panel and 1 uncoated panel placed together in channel: • 3 of AL1a (6 panels total) • 3 of AL1b (6 panels total)
Experimental Control Specimens	One (1) experimental control specimen consists of two (2) uncoated panels together in an aluminum channel. For each group of specimens placed in the oven or humidity chamber, the following will also be placed in the oven or humidity chamber: • 1 of AL1a (2 panels total, both uncoated) • 1 of AL1b (2 panels total, both uncoated)
Acceptance Criteria	No discoloration, pitting, white deposits, or other evidence of corrosion greater than that observed on uncoated control specimens

Major or Unique Equipment

- Oven
- Humidity cabinet

Data Recording and Calculations

- Report three (3) separate thickness measurements per coated panel.
- Report condition of test panels and experimental control specimens.

3.2. Chromium Content

Application Categories - LG, HG, LS, HS, AD

Test Description

This test is an evaluation of the chromium content of the DFL as supplied.

Prepare samples of DFL cured according to the manufacturer's instructions, and evaluate the chromium content in accordance with ASTM D3718 - 85a (*Standard Test Method for Low Concentrations of Chromium in Paint by Atomic Absorption Spectroscopy*, approved May 31 and November 29, 1985, reapproved July 1991).

Method Synopsis: Prepare a specimen of liquid coating or dried film for analysis by dry ashing at 500°C followed by digestion with potassium permanganate and sulfuric acid in a polytetrafluoroethylene (PTFE)-lined acid decomposition vessel at an elevated temperature. Determine the chromium in the filtered digestion mixture by atomic absorption spectroscopy.

Rationale

Chromium is a hazardous material included in the EPA 17 list of target chemicals to be reduced/eliminated. The technical representatives agreed that it is necessary to ensure that chromium is not introduced while lead is being eliminated, and selected the acceptance criteria of less than 100 ppm chromium content based on their processing requirements.

Note: No Military Specifications currently exist that specify chromium content for dry film lubricants. However, MIL-P-85582B (*Primer Coatings: Epoxy, Waterborne*; issued May 23, 1994, amendment issued August 31, 1994) and MIL-P-23377G (*Primer Coatings: Epoxy, High-Solids*; issued September 30, 1994) both specify no detection of chromium for nonchromate based corrosion inhibitors.

Test Methodology

Parameters	Char using a hot plate, then place into a muffle furnace at 475 to 500°C until ashing is complete (no more than 2 hours)
Number and Type of Specimens per Candidate DFL	2 samples, each 3 grams of dried film
Experimental Control Specimens	None required
Acceptance Criteria	Chromium content below 100 ppm

Major or Unique Equipment

- Atomic absorption spectrophotometer
- Furnace capable of 500 ± 10°C

Data Recording and Calculations

- Concentration of chromium (Cr) in the nonvolatile portion of the DFL is:

  Cr (ppm) = (C x F x A x V)/(NV x S)

  where

  C = Concentration of chromium in the aspirated test solution, mg/mL
  F = Dilution factor from Section 9.7 of ASTM D3718 - 85a (volume diluted to/volume of aliquot)
  A = Mean percent ash as determined in Section 9.2.5 of ASTM D3718 - 85a
  V = Volume diluted to in section 9.6 of ASTM D3718 - 85a (50 or 100 ml)
  NV= Percent nonvolatile of paint sample (use 100 if sample was a dried film)
  S = Mass of ash, g

- Report concentration, sensitivity, and detection limit.

3.3. Cured Film Thickness Uniformity

Application Categories - LG, HG, LS, HS

Test Description

This is an evaluation of the dry film thickness uniformity of candidate DFLs.

Apply DFL to 3" x 6" x 0.050" thick (minimum) test panels as recommended by the DFL manufacturer and cure. Visually examine the panels under normal work lighting and 3X magnification, to verify that the applied DFL is a smooth film of uniform color, with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping.

Measure cured film thickness at ten (10) locations on each test panel in accordance with ASTM E376 - 89 (*Standard Practice for Measuring Coating Thickness by Magnetic-Field or Eddy-Current (Electromagnetic) Test Methods*, approved October 27, 1989) by an appropriate method, using a probe no larger than 0.5 inches in diameter. More specific methods are contained in ASTM B244 - 79 (*Standard Method for Measurement of Thickness of Anodic Coatings on Aluminum and of Other Nonconductive Coatings on Nonmagnetic Basis Metals with Eddy-Current Instruments*, approved February 12, 1979), ASTM D1400 - 87 (*Standard Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base*, approved May 29, 1987), ASTM B499 - 88 (*Standard Test Method for*

Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals, approved August 26, 1988), or ASTM D1186 - 87 (*Standard Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base*, approved May 29, 1987).

Rationale

The participants in the JG-PP/PEWG effort agreed that it is important that any candidate dry film lubricant can be applied in an even film of thickness from 0.3 to 0.8 mils. The group judged that the acceptance criteria stated below would suitably reflect that requirement.

Test Methodology

Parameters	Apply DFL as recommended by manufacturer
Number and Type of Specimens per Candidate DFL	3 of AL1c (DFL cure temperature $\leq$ 400°F) or 3 of ST2b (DFL cure temperature > 400°F)
Experimental Control Specimens	None required
Acceptance Criteria	No more than one thickness measurement per panel below 0.0003 inch (0.3 mil) and no more than one thickness measurement per panel above 0.0008 inch (0.8 mil)

Major or Unique Equipment

Appropriate equipment for measuring film thickness, such as eddy current instrument, etc.

Data Recording and Calculations

- Report measurement technique used, and sensitivity of method.
- Report ten (10) film thicknesses measured on each panel.

3.4. Dry Tape Adhesion

Application Categories - LG, HG, LS, HS, AD

Test Description

This test method covers a procedure for establishing adequacy of surface adhesion of a coating by applying pressure-sensitive tape over a scribed area of the coating.

Apply DFL to 3" x 6" test panels (AL1a panels 0.020" thick, ST2b panels 0.050" thick) as recommended by the DFL manufacturer, to a thickness of 0.3 to 0.8 mils, and cure. Verify the film thickness by measurement at three separate locations on each panel. Visually examine the panels under normal work lighting and 3X magnification, to verify that the applied DFL is a smooth film of uniform color, with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping.

Evaluate the adhesion of the cured film in accordance with Procedure A of ASTM D2510 - 83 (*Standard Test Method for Adhesion of Solid Film Lubricants*, approved March 25, 1983), except do not immerse the panel in water, and examine the test specimen at 10X magnification after removal of the tape.

> Method Synopsis: Scribe two parallel incisions one inch apart through the DFL in accordance with Procedure A of ASTM D2510 - 83. Place a piece of tape over the incisions and smooth down by passing a 4.5 pound roller over it once. Remove the tape rapidly at approximately an 180° angle. Inspect the test specimen both with unaided vision and at 10X magnification.

Rationale

The technical representatives agreed that a dry tape test is a reasonable method to quickly determine whether a candidate DFL adheres well enough to merit further testing.

Test Methodology

Parameters	Apply DFL as recommended by manufacturer
Number and Type of Specimens per Candidate DFL	3 of AL1a (DFL cure temperature $\leq$ 400°F) or 3 of ST2b (DFL cure temperature > 400°F)
Experimental Control Specimens	None required
Acceptance Criteria	No exposure of underlying substrate

Major or Unique Equipment

- 1 inch masking tape, 3M Company Type 250 or equivalent
- 4.5 pound roller
- Carbide tip scribe

Data Recording and Calculations

- Report three (3) separate thickness measurements per coated panel.
- Report condition of test panels.

3.5. Elevated Temperature Material Compatibility

Application Categories - LG, HG, LS, HS

Test Description

This test allows assessment of substrate degradation promoted by the DFL under high temperature conditions.

For each type of test specimen, one specimen will be coated with the candidate DFL, and one specimen will be uncoated and will act as an experimental control. Apply DFL to 1" x 0.5" test specimens (AL1c, CO2, NI2a, NI2b, ST4, and ST9a specimens all 0.050" thick, CO1 specimens 0.062" thick, MG specimens 0.032" thick, NI1 specimens 0.035" thick, and ST3 specimens 0.550" thick) as recommended by the DFL manufacturer, to a thickness of 0.3 to 0.8 mils, and cure. Verify the film thickness by measurement at three separate locations on each specimen. Visually examine the specimens under normal work lighting and 3X magnification, to verify that the applied DFL is a smooth film of uniform color, with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping.

Heat the test specimens according to the time and temperature specified in Test Methodology. Allow the panels to cool to room temperature. Examine the specimens metallographically (at 500X magnification in cross section).

Rationale

This test requirement and the acceptance criteria were developed through the consensus of the participants in the JG-PP/PEWG effort. The participants agreed that it is important to assess the tendency, if any, of a candidate DFL to promote corrosion at elevated temperatures. The elevated temperatures chosen for this test are based on the properties of the substrate materials.

Test Methodology

Parameters	Heat in oven for 9 ± 1 hours
	• **LG, LS:**
	  ➢ Group A, 400 ± 5°F
	  ➢ Group B, 750 ± 5°F
	• **HG, HS:**
	  ➢ Group C, 1050 ± 5°F
	  ➢ Group D, 1600 ± 5°F

Number and Type of Specimens per Candidate DFL	1 of each
	• Group A
	  ➢ AL1c
	  ➢ MG
	• Group B
	  ➢ ST3
	• Group C
	  ➢ ST4
	  ➢ ST9a
	• Group D
	  ➢ CO1
	  ➢ CO2
	  ➢ NI1
	  ➢ NI2a
	  ➢ NI2b

Experimental Control Specimens	1 of each, uncoated
	• Group A
	  ➢ AL1c
	  ➢ MG
	• Group B
	  ➢ ST3
	• Group C
	  ➢ ST4
	  ➢ ST9a
	• Group D
	  ➢ CO1
	  ➢ CO2
	  ➢ NI1
	  ➢ NI2a
	  ➢ NI2b

Acceptance Criteria	No substrate degradation exceeding by 0.0002 inches or more the degradation observed on the uncoated control specimens

Major or Unique Equipment

Oven (air circulating furnace)

Data Recording and Calculations

- Report three (3) separate thickness measurements per coated panel.
- Report condition of test panels.

3.6. Fastener Corrosion

Application Categories - HS

Test Description

This test allows assessment of the tendency of a candidate DFL to accelerate corrosion of fasteners at elevated temperatures.

Apply DFL to nuts of the sizes specified in Test Methodology as recommended by the DFL manufacturer and cure. Install coated nuts on uncoated bolts, running the nuts on until the locking mechanism is engaged and the torque is as specified in Test Methodology. Assemble uncoated nuts and bolts to be used as experimental control specimens; treat these control assemblies in the same manner as the assemblies coated with the candidate DFL. Place a test nut/bolt assembly and a control assembly in each crucible identified in Test Methodology, so approximately half of each assembly is exposed to the atmosphere. Place the crucibles in an oven as specified in Test Methodology. Note that the water in the salt solutions will evaporate while the crucibles are in the oven. Inspect the assemblies visually after 100, 250, and 500 hours (± 12 hours) of exposure. After the full exposure time, remove the assemblies from the oven and separate the nuts and bolts. Examine the nuts and bolts at 500X magnification.

Rationale

This test requirement and the acceptance criteria were developed through the consensus of the participants in the JG-PP/PEWG effort. The participants agreed that it is important to assess the tendency, if any, of a candidate DFL to accelerate corrosion of fasteners at elevated temperatures. Exposure of nut/bolt assemblies to elevated temperatures while coated with salt solutions is intended to provide corrosive conditions to allow evaluation of any corrosion differences between coated and uncoated specimens.

Test Methodology

Parameters	• Crucible contents: ➢ 5% (by mass) sodium chloride (NaCl) solution ➢ 5% (by mass) sodium sulfate (Na_2SO_4) solution ➢ Equal parts (by volume) 5% NaCl solution and 5% Na_2SO_4 solution • 1400 ± 25°F (649 ± 14°C) for 1,000 ± 24 hours		
Installation Torque (inch-pounds)			
Bolt Diameter (inches)	Bolt material		
	NI3	NI2a or ST9b	CO2
0.190	50	35	NA
0.375	400	260	510
0.5	900	600	1100
Number and Type of Specimens per Candidate DFL	3 each of the following (1 of each per crucible): • Nut and bolt both NI3 • Nut and bolt both NI2a • Nut and bolt both ST9b • Nut NI2a, bolt CO2		
Experimental Control Specimens	3 each of the following (1 of each per crucible), uncoated: • Nut and bolt both NI3 • Nut and bolt both NI2a • Nut and bolt both ST9b • Nut NI2a, bolt CO2		
Acceptance Criteria	No evidence of substrate corrosion greater than that of the uncoated control assemblies		

Note: There will be no 0.190 inch diameter combination of CO2 bolt and NI2a nut tested.

Major or Unique Equipment

- Oven (air circulating furnace)
- Crucibles

Data Recording and Calculations

Report condition of coated and uncoated nut/bolt assemblies after "soaking" at 1400°F.

3.7. Fluid Resistance

Application Categories - LG, HG, LS, HS

Test Description

This test allows assessment of the susceptibility of the DFL to degradation and/or loss of adhesion due to contact with the listed fluids.

Apply DFL to 3" x 6" test panels (AL1a panels 0.020" thick, ST2a panels 0.050" thick) as recommended by the DFL manufacturer, to a thickness of 0.3 to 0.8 mils, and cure. Verify the film thickness by measurement at three separate locations on each panel. Visually examine the panels under normal work lighting and 3X magnification, to verify that the applied DFL is a smooth film of uniform color, with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping.

Partially immerse test panels in fluids as specified in Test Methodology and in accordance with ASTM D2510 - 83 (*Standard Test Method for Adhesion of Solid Film Lubricants*, approved March 25, 1983), Procedure C.

> Method Synopsis: Partially immerse panels for 24 $\pm$ 0.25 hours in specified fluid at 74 $\pm$ 2°F. Drain the panels for one (1) hour, triple-rinse in appropriate solvent listed below, and air dry at room temperature for one (1) hour. Examine visually and test adhesion of the DFL by a tape adhesion test in accordance with ASTM D2510 - 83, Procedure C. Examine panels at 4X magnification after removing tape. A powdery residue clinging to the tape is not sufficient reason for rejection.

Rationale

This test requirement and the acceptance criteria are as specified in MIL-L-46010E (*Lubricant, Solid Film, Heat Cured, Corrosion Inhibiting*; issued April 11, 1997) and MIL-L-46147B (*Lubricant, Solid Film, Air Cured (Corrosion Inhibiting)*; issued December 2, 1994). The fluids used for this test are considered representative of the possible exposure environments for DFLs in the indicated application categories, and were selected by consensus of the JG-PP/PEWG participants. In the interest of streamlining the test, not all fluids that are used in and around the affected engines have been selected for use in this test.

Test Methodology

Immersion Fluid	Reference Document	Rinse Solvent
Aircraft Turbine Engine Lubricating Oil, Synthetic Base	MIL-L-23699E	aliphatic naphtha
Anti-Icing Fluid (ethylene glycol/propylene glycol, 3:1 v/v)	MIL-A-8243D	distilled water
Aviation Turbine Fuel, JP-5	MIL-T-5624R	aliphatic naphtha
Aviation Turbine Fuel, JP-8	MIL-T-83133D	aliphatic naphtha
Distilled Water, filtered through 0.45 μm membrane (Type III)	ASTM D1193 - 91	none needed
Skydrol® 500B-4 Fire Resistant Hydraulic Fluid (Monsanto Company)	None	aliphatic naphtha
Silicone Base Damping Fluid (Dimethyl polysiloxane)	VV-D-1078B	aliphatic naphtha
Substitute Ocean Water	ASTM D1141 - 90	distilled water
Low Temperature Fire Resistant Aircraft and Missile Hydraulic Fluid, Synthetic Hydrocarbon Base	MIL-H-87257	aliphatic naphtha

Test Methodology (Part 2)

Parameters	Immersion in specified fluid at 74 $\pm$ 2°F (23 $\pm$ 1°C) for 24 $\pm$ 0.25 hours
Number and Type of Specimens per Candidate DFL	For each specified fluid: 3 of AL1a (DFL cure temperature $\leq$ 400°F) or 3 of ST2a (DFL cure temperature > 400°F)
Experimental Control Specimens	None required
Acceptance Criteria	No lifting, softening, blistering, cracking, peeling, significant discoloration, or loss of adhesion

Major or Unique Equipment

- Masking tape, code no. 250 (3M) or equivalent, 1 inch wide
- 4.5 pound rubber-covered roller, approximately 3.5 inches in diameter by 1.75 inches in width, surface of which has a Shore “A” durometer hardness value within the range of 70 to 80

Data Recording and Calculations

- Report three (3) separate thickness measurements per coated panel.
- Report condition of test panels.

3.8. Lead and Cadmium Content

Application Categories - LG, HG, LS, HS, AD

Test Description

This test is an evaluation of the lead and cadmium content of the DFL as supplied. The measurement of lead concentration when antimony pigments are also present may result in erroneous measurements.

Evaluate the lead and cadmium content of a DFL sample in accordance with ASTM D3335 - 85a (*Standard Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy*, approved July 16 and November 29, 1985).

> Method Synopsis: Prepare a specimen of the liquid coating or dried film for analysis by dry ashing. Prepare an acid extract of the ash. Measure the lead or cadmium content of the extract by atomic absorption spectroscopy.

Rationale

Lead is the hazardous material intended to be eliminated or reduced through this JG-PP/PEWG DFL project, and the technical representatives decided that measurement of lead content is necessary to verify that the candidate DFL truly is lead free. The participants decided to limit lead content of candidate DFL alternatives to 100 ppm maximum because at least one of the OEMs operates under a company policy that excludes products with higher lead content. Cadmium is also a hazardous material of concern, and the JG-PP/ PEWG participants agreed that it is necessary to ensure that high lead content is not being replaced by high cadmium content. The acceptance criteria of no more than 100 ppm cadmium was also chosen based on operating policy of at least one of the OEMs.

> Note: While the Qualified Products List for at least one current Military Specification for DFLs contains products designated as lead free, no current Military Specification for DFLs defines lead free. However, a maximum lead concentration of 0.06 percent by weight is defined as lead free in TT-P-664D (*Primer Coating, Alkyd, Corrosion-Inhibiting, Lead and Chromate Free, VOC-Compliant,* issued September 1, 1988).

Test Methodology

Parameters	Char using a hot plate, then place into a furnace at 475 to 500°C until ashing is complete (no more than 2 hours)
Number and Type of Specimens per Candidate DFL	2 samples, each 1 to 2 grams of dried film.
Experimental Control Specimens	None required
Acceptance Criteria	No more than 100 ppm lead or cadmium

Major or Unique Equipment

- Atomic absorption spectrophotometer
- Oven or Furnace capable of 500 ± 10°C

Data Recording and Calculations

- Concentration of heavy metal (C) in the DFL as a liquid is:

  C (ppm) = (C x F x 5000)/ (NV x S)

  where

  C = Concentration of lead or cadmium in the aspirated test solution, mg/mL
  F = Dilution factor from Section 9.9 of ASTM D 3335 - 85a (volume diluted to/volume of aliquot)
  5000 = Constant derived from multiplying the 50 mL volume obtained in procedure in Section 9.8 of ASTM D3335 - 85a by 100 (to convert NV used to a whole number) and 10^6 (to obtain ppm), then dividing by 10^6 (to convert grams of sample to mg)
  NV = Percent nonvolatile of paint sample (use 100 if sample was a dried film)
  S = Mass of specimen, g

- Report concentration, sensitivity, and detection limit.

3.9. Reciprocating Sliding Wear

Application Categories - LG, HG

Note: Where LG is indicated for testing, DFLs that are candidates for only LG applications or for both LG and HG applications should be tested. Where HG is indicated for testing, DFLs that are candidates for only HG applications or for both LG and HG applications should be tested.

Test Description

This test evaluates both the static and kinetic coefficients of friction and the wear behavior of cured DFL.

Use ASTM G115 - 93 (*Standard Guide for Measuring and Reporting Friction Coefficients*, approved March 15, 1993) as a guide in performing this evaluation. Calibrate all load cells, monitoring instruments, and other data monitoring devices.

Both the Fretting Wear and the Galling Wear procedures described below should be performed by the same testing facility, to ensure uniformity of results.

3.9.1. Fretting Wear

Prepare shoe and block specimens of identical materials, using either the shoe and block specimen described in Figure 1 (Specimen Configuration A), or the shoe described in Figure 2 and the block described in Figure 3 (Specimen Configuration B). Note that a single configuration (either A or B) must be used for all trials.

Apply DFL to a shoe test specimen of a substrate specified in Test Methodology and cure as recommended by the DFL manufacturer. Any LG category candidate alternative must be applied to a film thickness of 0.9 to 1.1 mils, and any HG category candidate alternative must be applied to a film thickness of 0.7 to 0.9 mils. Verify the film thickness by measurement at one or more location on each shoe specimen. Visually examine the panels under normal work lighting and 3X magnification, to verify that the applied DFL is a smooth film of uniform color, with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping.

Load the shoe and block specimens into a testing apparatus such as that shown in Figure 4, so that the wear surfaces are parallel to each other and to the direction of motion. Verify the positioning of the wear surfaces, such as by use of a pressure sensitive film. Start the reciprocating stroking with no contact force applied; increase contact pressure gradually to the

value specified in Test Methodology. The contact pressure should be ramped up proportionally over the first 3,000 cycles. The frequency and displacement of each stroke should be as specified in Test Methodology. The displacement listed in the test methodology is the distance from peak to peak traversed by the rig. Verify the stroke length using an appropriate method, such as traveling microscope or linear variable differential transducer (LVDT).

Report the specimen configuration used, and measure and report the beginning and end contact areas. Monitor and record (with an X-Y plotter or digital data acquisition) the normal and frictional forces during the test (at least at every 2^x cycles after full load is applied, where x is an integer).

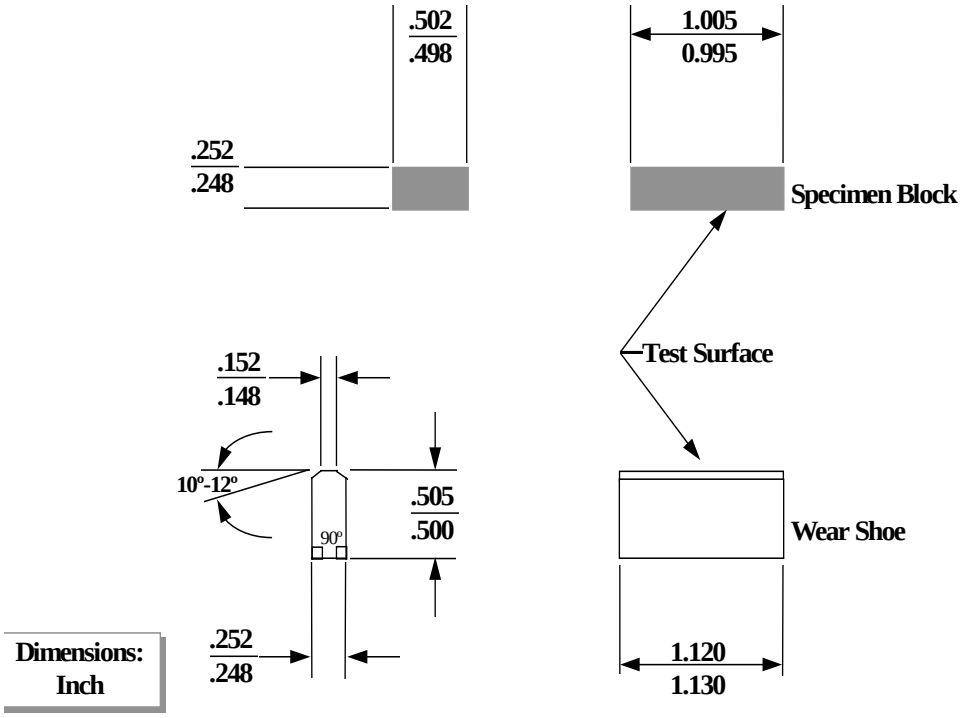
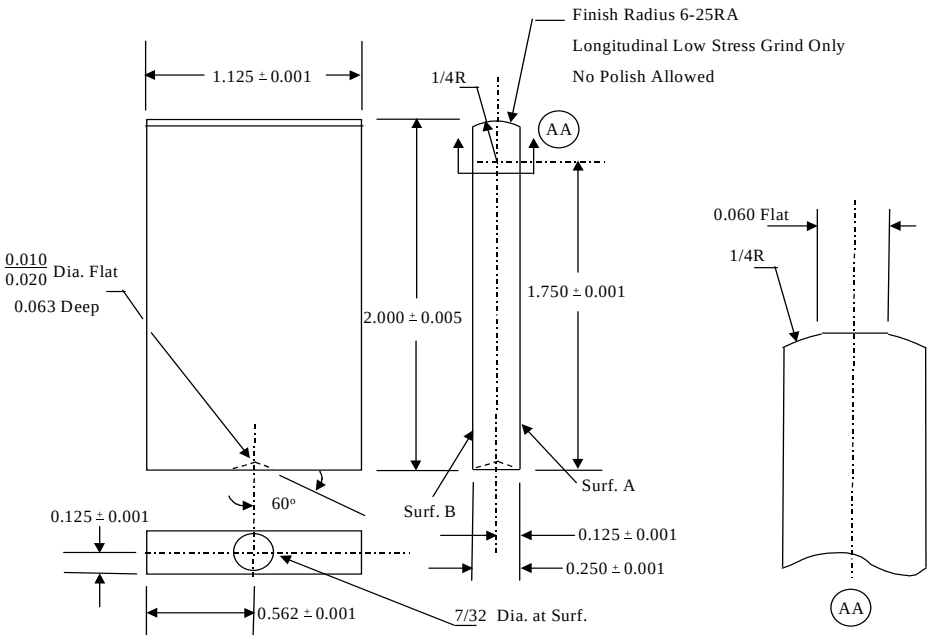
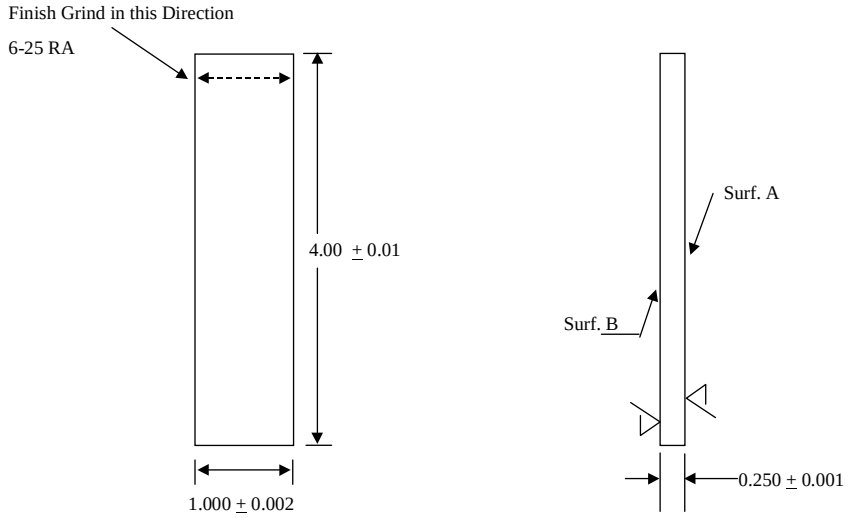


Figure 1. Specimen Configuration A - Shoe and Block Specimens



For both shoe and block specimens, surface A and surface B must be parallel to ± 0.001 total indicated runout.

Figure 2. Specimen Configuration B - Shoe Specimen



For both shoe and block specimens, surface A and surface B must be parallel to ± 0.001 total indicated runout.

Figure 3. Specimen Configuration B - Block Specimen

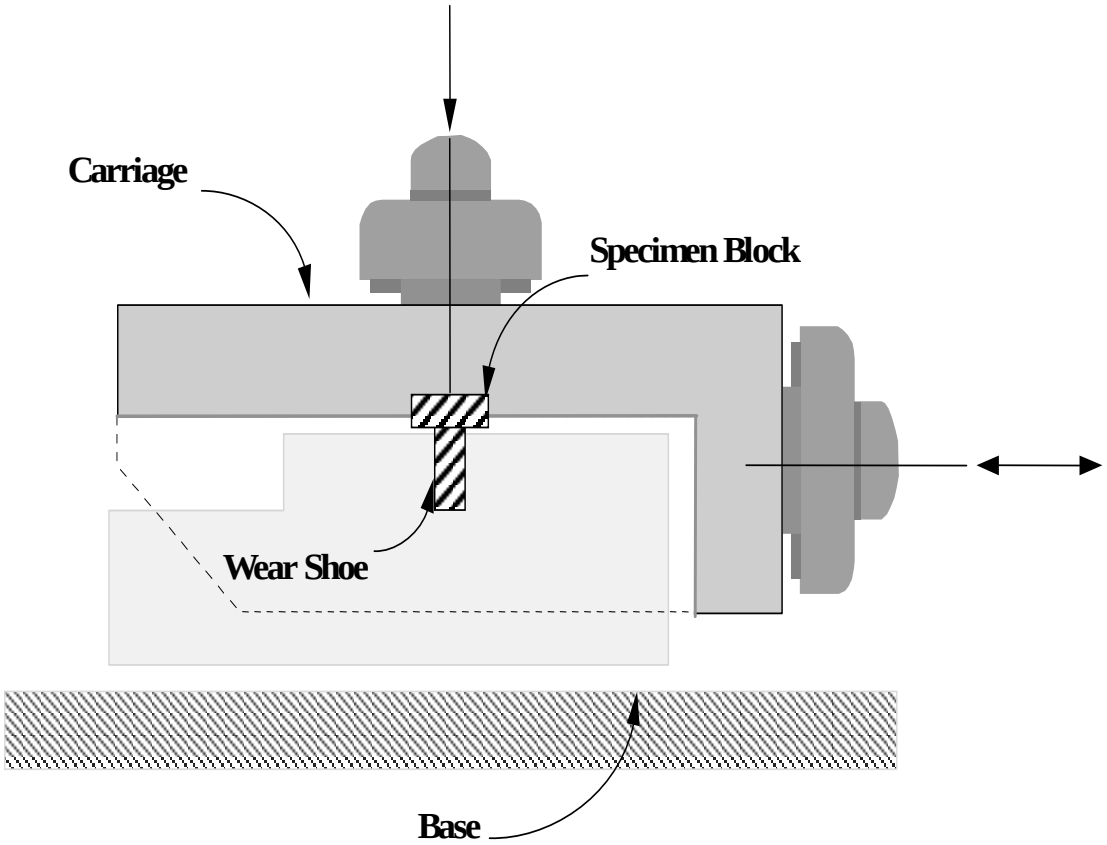


Figure 4. Typical Sliding Wear Test Apparatus

Rationale

This requirement, the test method, and the acceptance criteria were defined by consensus of the technical representatives (TRs) participating in the JG-PP/PEWG joint effort. The test conditions were chosen to simulate the conditions that cause fretting wear, and the test temperatures were chosen to allow subdivision of candidate DFLs into narrower operating temperature ranges than provided by the major application categories. The experimental control specimens coated with a currently accepted lead-containing DFL will allow verification of the results when compared to existing data on lead-containing DFLs.

Test Methodology

Parameters	Stroke Frequency*	60 cycles per second
	Stroke Displacement (peak to peak)	0.002 $\pm$ 0.001 inch
	Intermediate Inspections	every 250,000 cycles
	Test Duration	10^6 cycles

(Test Methodology table continued on next page)

Test Methodology (continued)

Contact Pressure and Temperature Conditions				
These contact pressure and temperature conditions will be applied to fretting evaluations of candidate DFLs intended for LG and HG applications as specified below. Note that the contact pressures should be 5 ± 1 ksi, 50 ± 5 ksi, and 85 ± 5 ksi.				
		Contact Pressure**		
Temperature	**Substrates**	**5 ksi**	**50 ksi**	**85 ksi**
75 ± 10°F	ST10, TI1a, TI1b	LG, HG	LG, HG	LG, HG
350 ± 25°F	ST10, TI1a, TI1b	LG, HG	LG, HG	LG, HG
600 ± 25°F	ST10, TI1a, TI1b	LG, HG	LG, HG	LG, HG
850 ± 25°F	ST10, TI1a	LG, HG	LG, HG	LG, HG
Number of Trials per Candidate DFL	At each temperature, perform three (3) separate trials at 5 ± 1 ksi, 50 ± 5 ksi, and 85 ± 5 ksi contact pressure using each one of the substrate materials specified above; shoe and block both of the same material, new shoe and block for each trial.			
Experimental Control Specimens	• Using a "low temperature" (intended for use up to 850°F) lead-containing DFL: Perform three (3) separate trials at each of the temperature and contact pressure conditions specified for LG DFLs using a shoe and block both of the same material (ST10, TI1a, and TI1b), new shoe and block for each trial. • Using a "high temperature" (intended for use from 850°F up to 1400°F) lead-containing DFL: Perform three (3) separate trials at each of the temperature and contact pressure conditions specified for HG DFLs using a shoe and block both of the same material (ST10, TI1a, and TI1b), new shoe and block for each trial.			
Acceptance Criteria: Wear Resistance	Residual film of lubricant with smooth or slightly striated wear pattern remaining on block and shoe specimens; no DFL flaking, base metal wear, or other signs of degradation			
Acceptance Criteria: Coefficient of Kinetic Friction	• LG: less than 0.12 • HG: less than 0.15			

* One cycle is one stroke forward and one backward

** 1 ksi equals 1,000 pounds force per square inch

Major or Unique Equipment

Reciprocating Sliding Wear Test Apparatus (shown in Figure 4) or equivalent

Data Recording and Calculations

- Report specimen configuration used.
- Report film thickness measurement(s) for each specimen.
- Report beginning and ending contact areas for each shoe and block.
- Coefficient of Kinetic Friction = Driving Force (F)/Contact Force (N) during motion (stroke)
- After the test, strip the shoe and examine visually and metallographically. Further destructive techniques such as mounting and polishing to verify coating integrity may be requested based on visual examination.

3.9.2. Galling Wear

Prepare shoe and block specimens of identical materials, using either the shoe and block specimen described in Figure 1 (Specimen Configuration A), or the shoe described in Figure 2 and the block described in Figure 3 (Specimen Configuration B). Note that a single configuration (either A or B) must be used for all trials.

Apply DFL to a shoe test specimen of a substrate specified in Test Methodology and cure as recommended by the DFL manufacturer. Any LG category candidate alternative must be applied to a film thickness of 0.9 to 1.1 mils, and any HG category candidate alternative must be applied to a film thickness of 0.2 to 1.0 mils. Verify the film thickness by measurement at one or more locations on each shoe specimen. Visually examine the panels under normal work lighting and 3X magnification, to verify that the applied DFL is a smooth film of uniform color, with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping.

Load the shoe and block specimens into a testing apparatus such as that shown in Figure 4, so that the wear surfaces are parallel to each other and to the direction of motion. Verify the positioning of the wear surfaces, such as by use of a pressure sensitive film. Start the reciprocating stroking with no contact force applied; increase contact pressure gradually to the value specified in Test Methodology. The contact pressure should be ramped up proportionally over the first 50 cycles. The frequency and displacement of each stroke should be as specified in Test Methodology. The displacement listed in the test methodology is the distance from peak

to peak traversed by the rig. Verify the stroke length using an appropriate method, such as traveling microscope or linear variable differential transducer (LVDT).

Report the specimen configuration used, and measure and report the beginning and end contact areas. Monitor and record (with an X-Y plotter or digital data acquisition) the normal and frictional forces during the test (at least at every 2^x cycles after full load is applied, where x is an integer).

Rationale

This requirement, the test method, and the acceptance criteria were defined by consensus of the technical representatives (TRs) participating in the
JG-PP/PEWG joint effort. The test conditions were chosen to simulate the conditions that cause galling wear, and the test temperatures were chosen to allow subdivision of candidate DFLs into narrower operating temperature ranges than provided by the major application categories. The experimental control specimens coated with a currently accepted lead-containing DFL will allow verification of the results when compared to existing data on lead-containing DFLs.

Test Methodology

Parameters	Stroke Frequency*	1 cycle per second	
	Stroke Displacement (peak to peak)	0.01 ± 0.001 inch	
	Intermediate Inspections	every 2,500 cycles	
	Test Duration	10^4 cycles (except where noted**)	
Contact Pressure and Temperature Conditions			
These contact pressure and temperature conditions will be applied to galling evaluations of candidate DFLs intended for LG and HG applications as specified below. Note that the contact pressures should be 50 ± 5 ksi and 85 ± 5 ksi.			
		Contact Pressure* **	
Temperature	**Substrates**	**50 ksi**	**85 ksi**
75 ± 10°F	ST10, TI1a, TI1b, NI3** (HG only)	LG, HG	LG, HG
350 ± 25°F	ST10, TI1a, TI1b	LG, HG	LG, HG
600 ± 25°F	ST10, TI1a, TI1b	LG, HG	LG, HG
850 ± 25°F	ST10, TI1a	LG, HG	LG, HG

(Test Methodology table continued on next page)

Test Methodology (continued)

Temperature	Substrates	Contact Pressure***	
		50 ksi	85 ksi
900 ± 25°F	NI3**	HG	
Number of Trials per Candidate DFL	At each specified temperature and contact pressure, perform three (3) separate trials using each one of the substrate materials specified above; shoe and block both of the same material, new shoe and block for each trial.		
Experimental Control Specimens	• Using a "low temperature" (intended for use up to 850°F) lead-containing DFL: Perform three (3) separate trials at each of the temperature and contact pressure conditions specified for LG DFLs using a shoe and block both of the same material (ST10, TI1a, and TI1b), new shoe and block for each trial • Using a "high temperature" (intended for use from 850°F up to 1400°F) lead-containing DFL: Perform three (3) separate trials for the temperature and contact pressure conditions specified for HG DFLs, using a shoe and block both of the same material (ST10, TI1a, TI1b, and NI3), new shoe and block for each trial.		
Acceptance Criteria: Wear Resistance	Residual film of lubricant with smooth or slightly striated wear pattern remaining on block and shoe specimens; no DFL flaking, base metal wear, or other signs of degradation		
Acceptance Criteria: Coefficient of Kinetic Friction	• LG: less than 0.12 • HG: less than 0.15		

* One cycle is one stroke forward and one backward

** For NI3, the test duration should be 5,000 cycles for each trial

*** 1 ksi equals 1,000 pounds force per square inch

Major or Unique Equipment

Reciprocating Sliding Wear Test Apparatus (shown in Figure 4) or equivalent

Data Recording and Calculations

- Report specimen configuration used.
- Report film thickness measurement(s) for each specimen.

- Report beginning and ending contact areas for each shoe and block.
- Coefficient of Kinetic Friction = Driving Force (F)/Contact Force (N) during motion (stroke)
- After the test, strip the shoe and examine visually and metallographically. Further destructive techniques such as mounting and polishing to verify coating integrity may be requested based on visual examination.

3.10. Salt Spray (Fog) Corrosion Resistance

Application Categories - LG

Test Description

This test allows evaluation of the ability of a candidate DFL to form a barrier that prevents corrosion of phosphated carbon steel coated with the DFL and exposed to salt spray.

Apply DFL to 3" x 6" x 0.063" test specimens (panels) as recommended by the DFL manufacturer, to a thickness of 0.3 to 0.8 mils, and cure. Verify the film thickness by measurement at three separate locations on each panel. Visually examine the panels under normal work lighting and 3X magnification, to verify that the applied DFL is a smooth film of uniform color, with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping.

Place test panels in a salt spray chamber operated in accordance with ASTM B117 - 94 (*Standard Practice for Operating Salt Spray (Fog) Testing Apparatus*, approved February 15, 1994). Remove the panels from the salt spray chamber after 100 hours of exposure and visually examine the test panels after rinsing and drying in accordance with ASTM D1654 - 92 (*Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments*, approved October 15, 1992), Method 1 (Air Blow-Off).

Rationale

This test of corrosion resistance of phosphated carbon steel coated with the candidate DFL and the acceptance criteria match the requirements specified in MIL-L-46010E (*Lubricant, Solid Film, Heat Cured, Corrosion Inhibiting*; issued April 11, 1997) and MIL-L-46147B (*Lubricant, Solid Film, Air Cured (Corrosion Inhibiting)*; issued December 2, 1994). The JG-PP/PEWG participants agreed that this test is necessary to qualify candidate DFLs for use on steel alloys not resistant to corrosion, which are used in some of the weapon systems affected by this JTP.

Test Methodology

Parameters	5% NaCl solution sprayed at 35°C, for 100 hours
Number and Type of Specimens per Candidate DFL	3 of ST1
Experimental Control Specimens	None required
Acceptance Criteria	No more than three (3) corrosion spots per specimen and no corrosion spots larger than 1.0 millimeter diameter

Major or Unique Equipment

Salt spray chamber

Data Recording and Calculations

- Report three (3) separate thickness measurements per coated panel.
- Report condition of test panels.

3.11. Solvent Rub

Application Categories - LG, HG, LS, HS

Test Description

This test allows assessment of the resistance of the cured DFL to removal or loss of film adhesion due to exposure to solvent.

Apply DFL to 3" x 6" test panels (AL1a panels 0.020" thick, ST2b panels 0.050" thick) as recommended by the DFL manufacturer, to a thickness of 0.3 to 0.8 mils, and cure. Verify the film thickness by measurement at three separate locations on each panel. Visually examine the panels under normal work lighting and 3X magnification, to verify that the applied DFL is a smooth film of uniform color, with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping.

Soak one cotton swab in each fluid specified in Test Methodology and rub each swab vigorously across the panel surface for 60 seconds. Do not apply more than one fluid to any panel. Examine the test panels at 4X magnification. Presence of lubricant on cotton swab is not reason for rejection.

Rationale

Vigorous rubbing with MEK or acetone is a common test for assessing the quality of the cure of organic coatings. The participants in the JG-PP/PEWG joint project agreed that the four solvents below represent the most likely cleaning fluids used around engines, and that this solvent rub test is necessary to verify that candidate DFLs will not be inadvertently removed during engine cleaning.

Test Methodology

Parameters	Rub vigorously for 60 ± 5 seconds with each of the following: • Methyl ethyl ketone (MEK) • Acetone • Degreasing solvent (P-D-680B, Type II) • Isopropyl alcohol
Number and Type of Specimens per Candidate DFL	For each specified solvent: 3 of AL1a (DFL cure temperature ≤ 400°F) or 3 of ST2b (DFL cure temperature > 400°F)
Experimental Control Specimens	None required
Acceptance Criteria	No separation of lubricant film or exposure of substrate

Major or Unique Equipment

Cotton swabs

Data Recording and Calculations

- Report three (3) separate thickness measurements per coated panel.
- Report condition of test panels.

3.12. Stress Corrosion

Application Categories - LG, LS, AD

Test Description

This test allows evaluation of the tendency of the candidate DFL to cause stress corrosion cracking of titanium alloys.

Perform the evaluation as described in ASTM F945 - 85 (*Standard Test Method for Stress-Corrosion of Titanium Alloys by Aircraft Engine Cleaning Materials*, approved July 26, 1985, reapproved 1993), Method A, with the modifications described below. In this test, titanium specimens are bent and restrained to a "U" shape and heated, after being dipped in selected chemical solutions.

Prepare 5.6" x 0. 5" x 0.050" thick test specimens rather than the 3" x 0.75" x 0.050" thick specimens described in ASTM F945 - 85. For the purposes of this test, the titanium specimens may be restrained with an appropriate clip rather than a bolt. Instead of the procedure specified in section 8.1.3 of ASTM F945 - 85 (immersion in selected solution), coat three (3) restrained test specimens with the candidate DFL as recommended by the DFL manufacturer, to a thickness of 0.3 to 0.8 mils, and cure the DFL according to the manufacturer's instructions. Verify the film thickness by measurement at three separate locations on each specimen. Visually examine the specimens under normal work lighting and 3X magnification, to verify that the applied DFL is a smooth film of uniform color, with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping.

Place the restrained test specimens and experimental control specimens in an air circulating furnace as specified in Test Methodology. After removing the specimens from the furnace, allow them to cool to room temperature then examine the specimens at 10X magnification as described in Sections 8.3 through 9.3.3 of ASTM F945 - 85. Note that some experimental control specimens are heated without being coated, to establish the acceptability of the titanium alloy material used for the specimens, and some specimens are heated after being dipped in a salt solution, to establish the sensitivity to stress corrosion attack of the titanium alloy material used for the specimens.

Rationale

This test is an accepted industry standard for assessing a material's tendency to cause stress corrosion cracking of titanium alloys. The participants in the JG-PP/PEWG effort agreed that this test is necessary to prevent use of any candidate DFL in a situation in which it would cause stress corrosion cracking of titanium alloys. The use of specimens coated with a currently accepted lead-containing DFL will allow the assessment of the validity of applying this test method, because current military DFL specifications do not require any stress corrosion cracking tests.

Test Methodology

Parameters	Heat at 900 ± 20°F (480 ± 10°C) for 8 ± 0.2 hours
Number and Type of Specimens per Candidate DFL	3 of TI1a 3 of TI2
Experimental Control Specimens	3 of TI1a, uncoated 3 of TI1a, dipped in 3% (by mass) sodium chloride solution) 3 of TI1a, coated with a currently accepted lead-containing DFL 3 of TI2, uncoated 3 of TI2, dipped in 3% (by mass) sodium chloride solution) 3 of TI2, coated with a currently accepted lead-containing DFL
Acceptance Criteria	No cracking of substrate

Major or Unique Equipment

- Press forming apparatus
- Air circulating furnace

Data Recording and Calculations

- Report three (3) separate thickness measurements per coated specimen.
- Report condition of test specimens.

3.13. Sulfurous Acid Salt Spray

Application Categories - LG, AD

Test Description

This test allows accelerated evaluation of the corrosion characteristics of steel panels coated with the DFL, through exposure to a sprayed solution of sulfurous acid and substitute ocean water.

Prepare 3" x 6" ST10 test specimens. Apply DFL as recommended by the DFL manufacturer, to a thickness of 0.3 to 0.8 mils, and cure. Verify the film thickness by measurement at three separate locations on each panel. Visually examine the panels under normal work lighting and 3X magnification, to verify that the applied DFL is a smooth film of uniform color, with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping.

Place test panels in a salt spray chamber operated in accordance with ASTM B117 - 94 (*Standard Practice for Operating Salt Spray (Fog) Testing Apparatus*, approved February 15, 1994). Expose specimens to a spray of synthetic sea water and sulfurous acid as specified in Test Methodology. Spray formula should consist of 900 mL synthetic sea water (mixed in accordance with ASTM D1141-90, *Standard Specification for Substitute Ocean Water,* 41.95 g/L) mixed with 2 mL sulfurous acid (not less than 6.0% assay as sulfur dioxide). At the end of the required exposure time, visually examine the test specimens.

Rationale

The combination of salt and sulfurous acid in the spraying solution, along with the cycling effect of solution spray followed by drying, creates a severe corrosion environment that allows rapid evaluation of the corrosion protection qualities of the candidate DFL. The Navy requires this evaluation because of the high potential for its weapon systems to be exposed to sea air simultaneously with SO_2 air pollution and/or "acid rain." The Air Force also deploys some weapon systems in regions susceptible to these severe conditions. This test and the acceptance criteria match the requirements specified in MIL-L-46010E (*Lubricant, Solid Film, Heat Cured, Corrosion Inhibiting*; issued April 11, 1997).

Test Methodology

Parameters	Four (4) cycles of • 2 hours synthetic sea water and sulfurous acid spray followed by • 24 hours air dry at room temperature
Number and Type of Specimens per Candidate DFL	3 of ST10
Experimental Control Specimens	None required
Acceptance Criteria	No pitting, staining, or other visible evidence of corrosion

Major or Unique Equipment

- Salt spray chamber

Data Recording and Calculations

- Report three (3) separate thickness measurements per coated specimen.
- Report condition of test specimens.

3.14. Thermal Shock Stability

Application Categories - LG, HG, LS, HS

Test Description

This test assesses the adhesion of the DFL to the substrate after exposure to wide swings in ambient temperature, and helps in estimation of the durability of the DFL in use in engines, where repeated heating and cooling cycles occur.

Apply DFL to 3" x 6" x 0.050" thick test panels as recommended by the DFL manufacturer, to a thickness of 0.3 to 0.8 mils, and cure. Verify the film thickness by measurement at three separate locations on each panel. Visually examine the panels under normal work lighting and 3X magnification, to verify that the applied DFL is a smooth film of uniform color, with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping.

Expose test panels to a temperature cycle as specified in Test Methodology, and using ASTM D2511 - 83 (*Standard Test Method for Thermal Shock Sensitivity of Solid Film Lubricants*, approved March 25, 1983) Section 9 as a guide. Wipe any condensation from the panels with a lint-free cloth, then examine the test panels visually. Test adhesion of the DFL by a tape adhesion test in accordance with ASTM D2510 - 83 (*Standard Test Method for Adhesion of Solid Film Lubricants*, approved March 25, 1983), Procedure B.

Rationale

The test and the acceptance criteria parallel the requirements specified in MIL-L-46010E (*Lubricant, Solid Film, Heat Cured, Corrosion Inhibiting*; issued April 11, 1997) and MIL-L-46147B (*Lubricant, Solid Film, Air Cured (Corrosion Inhibiting)*; issued December 2, 1994), except that different temperatures are used in this test, to correspond to the operating requirements identified by the participants. Dry film lubricants are often used in circumstances where wide temperature swings occur. The participants in the JG-PP/PEWG effort agreed that this test will help in elimination of DFLs which are unsuitable for the required applications. The use of specimens coated with a currently accepted lead-containing DFL will allow the assessment of the validity of applying this test method, because the temperatures have been modified from those used in the military specifications.

Test Methodology

Parameters	• One (1) temperature cycle:
	• Hold panel(s) at the temperature specified below for three (3) hours, then immediately place panel(s) in cold chamber at -65 ± 5°F (-54 ± 3°C) and hold for an additional three (3) hours.
	
	• LG, LS
	  ➢ 600 ± 10°F
	  ➢ 900 ± 10°F
	• HG, HS
	  ➢ 1200 ± 25°F
	  ➢ 1400 ± 25°F

Number and Type of Specimens per Candidate DFL	First holding temperature (upper temperature):
	• 600 ± 10°F
	  ➢ 3 of TI1b
	  ➢ 3 of ST5
	• 900 ± 10°F
	  ➢ 3 of ST5
	• 1200 ± 25°F
	  ➢ 3 of NI2a
	• 1400 ± 25°F
	  ➢ 3 of NI2a

Experimental Control Specimens	All control specimens described below should be coated with a currently accepted lead-containing DFL.
	
	First holding temperature (upper temperature):
	• 600 ± 10°F
	  ➢ 3 of TI1b
	  ➢ 3 of ST5
	• 900 ± 10°F
	  ➢ 3 of ST5
	• 1200 ± 25°F
	  ➢ 3 of NI2a
	• 1400 ± 25°F
	  ➢ 3 of NI2a

Acceptance Criteria	No flaking, cracking, softening, lifting, or loss of adhesion greater than that observed for the control (lead containing DFL)

Major or Unique Equipment

- Oven (air circulating furnace)
- Cold chamber (See ASTM D2511 - 83 requirements)
- Masking tape, code no. 250 (3M) or equivalent, 1 inch wide
- 4.5 pound rubber-covered roller, approximately 3.5 inches in diameter by 1.75 inches in width, surface of which has a Shore "A" durometer hardness value within the range of 70 to 80

Data Recording and Calculations

- Report three (3) separate thickness measurements per coated panel.
- Report condition of test specimens.

3.15. Thermal Stability by Simultaneous Differential Thermal Analysis-Thermogravimetric Analysis (SDT)

Application Categories - LG, HG, LS, HS

Test Description

This test permits an assessment of the useful operating temperature of a candidate DFL and the thermal stability of a candidate at specific temperatures.

For each combination of atmosphere, temperature ramping sequence, and ending temperature specified below in Test Methodology, place two (2) cured samples of the candidate DFL into the SDT sample chamber (it may be necessary to place the two samples in the chamber sequentially rather than simultaneously). For each evaluation, adjust the chamber atmosphere and increase the temperature of the sample chamber from room temperature as specified in Test Methodology. Measure and record chamber temperature, sample mass, and thermal response (whether exothermic or endothermic) continuously or at intervals no greater than every five minutes during the temperature increase. While samples are being held at the specified temperature, measure and record sample mass and thermal response at intervals no greater than every hour.

For each high temperature candidate DFL (HG and HS) and for each candidate DFL that is being considered for both low temperature and high temperature applications, ramp at 50°F per minute in air up to 1450°F and hold for 24 hours before performing any other analysis. If the candidate alternative DFL does not exhibit any significant degradation/reaction under these conditions, it may be presumed that no degradation/reaction would occur at any lower temperature. Under these circumstances, do not perform any other 50°F per minute ramp procedure on that candidate.

For each low temperature candidate DFL (LG and LS), ramp at 50°F per minute in air up to 900°F and hold for 24 hours before performing any other analysis. If the candidate alternative DFL does not exhibit any significant degradation/reaction under these conditions, it may be presumed that no degradation/reaction would occur at any lower temperature. Under these circumstances, do not perform any other 50°F per minute ramp procedure on that candidate.

Rationale

This test is intended to demonstrate the temperature stability of a candidate DFL for either low temperature use (up to 850°F) or high temperature use (up to 1400°F). Testing will be conducted using temperatures up to 50ºF above the maximum expected operating range (900°F for LG, LS and 1450°F for HG and HS) because ramping up to 50°F above the expected useful temperature allows evaluation of any detrimental effects that may occur very close to 850°F or 1400°F. The testing will be performed in air and in an inert atmosphere (argon or nitrogen). The use of the inert atmosphere will allow observation of thermal degradation, while testing in air will demonstrate the resistance of the DFL to oxidation. Holding each candidate DFL at an elevated temperature for 24 hours is intended to facilitate identification of any detrimental effects that may be caused by extended exposure to those temperatures.

Test Methodology

Parameters	In air and in nitrogen or argon:
	• Ramp temperature at 10°F per minute to holding temperature specified below, then halt test
	• Ramp temperature at 50°F per minute to holding temperature specified below, then hold at that temperature for 24 ± 1 hours, then halt test.
	
	Holding Temperatures:
	• **LG, LS**:
	  ➢ 600°F
	  ➢ 900°F
	• **HG, HS**:
	  ➢ 1200°F
	  ➢ 1450°F

(Test Methodology table continued on next page)

Test Methodology (continued)

Number and Type of Specimens per Candidate DFL	**2 samples** of each cured candidate DFL for each combination of atmosphere, temperature ramping sequence, and holding temperature. Note that when the temperature ramping rate is 10°F per minute, one set of samples heated to the maximum holding temperature will provide all required data. For instance, a HG or HS candidate DFL heated at a temperature ramping rate of 10°F per minute to 1450°F has also been heated to 1200°F.
Experimental Control Specimens	None required
Acceptance Criteria	This test is intended to provide baseline information on the temperature limits of a DFL. The temperature limits will define and categorize the DFL as low temperature (up to 850°F) or high temperature (between 850°F and 1400°F). The *useful temperature limit* will be defined as the temperature above 400°F at which there is a substantial change (increase or decrease) in the mass of the cured DFL sample.

Major or Unique Equipment

TA Instruments SDT 2920 Simultaneous DTA/TGA Device or equivalent

Data Recording and Calculations

Report chamber temperature, sample mass, and thermal response (whether exothermic or endothermic) data collected during temperature ramping and holding.

3.16. Torque-Tension Evaluation

Application Categories - LS, HS

Test Description

This procedure allows evaluation of the resistance of each candidate DFL to degradation during repeated installation and removal of fasteners, both at room temperature and when exposed to elevated temperatures.

Determine the relationship between angle of turn and clamping force for each bolt size as follows (room temperature calibration procedure):

Apply DFL to a self-locking nut as recommended by the DFL manufacturer. Note that if the manufacturer of a HS candidate DFL recommends the use of an assembly aid and/or disassembly aid, such an aid should be used. Measure and record the initial length of a matching, uncoated bolt to the nearest 0.0001 inch. Install the uncoated bolt through a shim (same material as the block fixture), a load cell, the hole of a block fixture, and the coated nut (see Figure 5). Tighten the nut, using a constant rate of turn and keeping the bolt in a fixed position. Continuously record force, torque, and the angle of turn using an X-Y plotter or real-time digital data acquisition system. Make note of the self-locking run-on torque and the grab torque. Compress the load cell to the clamping load specified in Test Methodology. Measure and record the assembly (clamp) torque and the assembled bolt length (to the nearest 0.0001 inch). Record force, torque, and angle of turn while loosening the nut. Pause briefly after turning each nut through a 180° angle from its clamped position. Continue to remove the nut, measuring and recording the breakaway torque and the self-locking run-off torque. After each nut has been removed from its bolt, measure and record the bolt length to the nearest 0.0001 inch.

Visually examine each nut and bolt, and record observations of any changes to each bolt or nut, especially any thread distortion, galling, or scratching. Generate a plot of clamping load versus angle of turn.

Repeat this sequence four more times using the same nut and bolt, for a total of five (5) assembly/disassembly cycles for each nut/bolt set.

Perform this room temperature calibration procedure with a total of nine (9) separate sets of nuts and bolts for each bolt size and combination of materials specified below in Test Methodology for room temperature evaluation. For each assembly/disassembly cycle, calculate an average angle of turn required to create the desired clamping force, using the values recorded for the nine separate nut/bolt sets. These calculated average angles of turn will be used to achieve the clamping force for the elevated temperature exposure procedure.

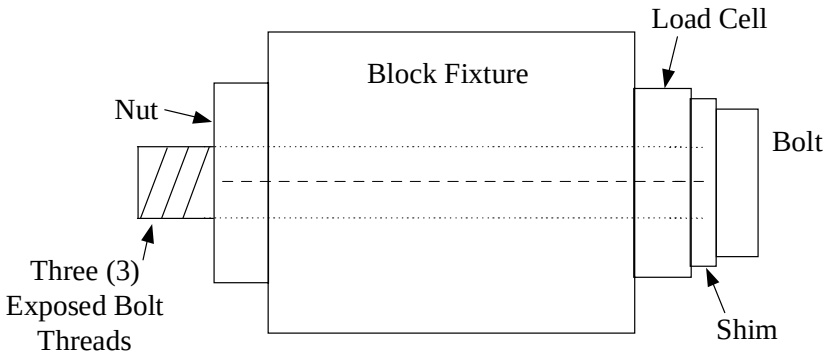


Figure 5. Clamping Force Calibration Apparatus

If the self-locking run-on torque, the self-locking run-off torque, or the ratio of breakloose torque to assembly torque calculated during the room temperature calibration procedure exceeds the value specified in the Test Methodology (acceptance criteria), do not test that material combination using any of the elevated temperatures.

Evaluate the effects of elevated temperature exposure on the antiseizing properties of each DFL by performing the following procedure:

For each combination of substrate materials specified in Test Methodology, apply DFL as recommended by the DFL manufacturer to nine (9) self-locking nuts of each size listed in Test Methodology. Note that if the manufacturer of a HS candidate, DFL recommends the use of an assembly aid and/or disassembly aid, such an aid should be used. Measure and record the initial lengths of matching, uncoated, bolts to the nearest 0.0001 inch. Install the nine uncoated bolts through a block fixture and tighten each coated nut to the angle of turn previously determined to create the specified clamping load. Note that these assemblies contain no load cells. Keep each bolt in a fixed position when tightening the nut. While installing each nut and bolt, measure and record the self-locking run-on torque and the grab torque. After each nut has been tightened to the specified angle of turn (determined during the calibration procedure), measure and record the assembly (clamp) torque and the assembled bolt length (to the nearest 0.0001 inch).

Heat the assembly as specified in Test Methodology. After the assembly has cooled to room temperature, loosen each nut while measuring and recording the breakloose torque. Pause briefly after turning each nut through a 180° angle from its clamped position. Continue to remove the nut, measuring and recording the breakaway torque and the self-locking run-off torque. After each nut has been removed from its bolt, measure and record the bolt length to the nearest 0.0001 inch. Visually examine

each nut and bolt, and record observations of any changes to each bolt or nut, especially any thread distortion, galling, or scratching.

Repeat the preceding test procedure for a total of five (5) assembly/disassembly cycles, being sure to match each bolt with the same nut for all cycles.

Rationale

The torque-tension curve achieved with any given nut-bolt assembly will depend upon the lubricating properties of the DFL applied. The calibration of clamping force with angle of turn during the room temperature evaluation is intended to allow creation of reproducible clamping forces with different DFLs when the assemblies are prepared for heating.

This test procedure was developed through consensus of the participants in the JG-PP/PEWG effort to simulate operational environments and allow evaluation of the antiseizing properties of candidate DFLs.

The measurement of loaded and unloaded bolt lengths will allow correlation of bolt strain with clamping force and torque.

Test Methodology

<table>
<tr><td rowspan="2">Parameters</td><td colspan="3">Heat after assembly, before disassembly:
• LS
➢ Room temperature - no time delay before removal
➢ 600 ± 25°F for 100 ± 8 hours
➢ 950 ± 25°F for 100 ± 8 hours
• HS
➢ Room temperature - no time delay before removal
➢ 950 ± 25°F for 100 ± 8 hours
➢ 1100 ± 25°F for 100 ± 8 hours
➢ 1400 ± 25°F for 100 ± 8 hours</td></tr>
<tr><td>Bolt Size (inches) - Thread Pitch</td><td>Torque increase indicating seating (inch-pounds)</td><td>Clamping Load (pounds)</td></tr>
<tr><td></td><td>0.250 - 28</td><td>10</td><td>2,355</td></tr>
<tr><td></td><td>0.500 - 20</td><td>25</td><td>11,250</td></tr>
</table>

(Test Methodology table continued on next page)

Test Methodology (Continued)

<table>
<tr><td>Number and Type of Specimens per Candidate DFL</td><td colspan="5">For each nut/bolt diameter specified above, nine (9) sets of nut and bolt per each combination of temperature and materials specified below; each block fixture should contain holes for nine (9) nut/bolt assemblies and should be of an appropriate thickness so that at least three (3) full threads are exposed after the specified angle of turn is achieved:
<table>
<tr><th colspan="2" rowspan="2">Heating Temperature</th><th colspan="3">Substrate Materials</th></tr>
<tr><th>Nut</th><th>Bolt</th><th>Block Fixture</th></tr>
<tr><td rowspan="8">Room Temp.</td><td>LS</td><td>ST9b</td><td>TI1a</td><td>TI1a</td></tr>
<tr><td>LS</td><td>ST9b</td><td>NI3</td><td>TI1a</td></tr>
<tr><td>LS, HS</td><td>ST9b</td><td>NI3</td><td>NI3</td></tr>
<tr><td>HS</td><td>NI2a</td><td>NI3</td><td>NI3</td></tr>
<tr><td>HS</td><td>NI2a</td><td>NI2a</td><td>NI2a</td></tr>
<tr><td>HS</td><td>NI3</td><td>NI2a</td><td>NI2a</td></tr>
<tr><td>HS</td><td>NI2a</td><td>NI3</td><td>NI2a</td></tr>
<tr><td rowspan="2">600 ± 25°F</td><td>LS</td><td>ST9b</td><td>TI1a</td><td>TI1a</td></tr>
<tr><td>LS</td><td>ST9b</td><td>NI3</td><td>TI1a</td></tr>
<tr><td rowspan="4">950 ± 25°F</td><td>LS, HS</td><td>ST9b</td><td>NI3</td><td>NI3</td></tr>
<tr><td>HS</td><td>NI2a</td><td>NI3</td><td>NI3</td></tr>
<tr><td>HS</td><td>NI3</td><td>NI2a</td><td>NI2a</td></tr>
<tr><td>HS</td><td>NI2a</td><td>NI3</td><td>NI2a</td></tr>
<tr><td>1100 ± 25°F</td><td>HS</td><td>ST9b</td><td>NI3</td><td>NI3</td></tr>
<tr><td rowspan="3"></td><td>HS</td><td>NI2a</td><td>NI3</td><td>NI3</td></tr>
<tr><td>HS</td><td>NI3</td><td>NI2a</td><td>NI2a</td></tr>
<tr><td>HS</td><td>NI2a</td><td>NI3</td><td>NI2a</td></tr>
<tr><td rowspan="3">1400 ± 25°F</td><td>HS</td><td>NI2a</td><td>NI2a</td><td>NI2a</td></tr>
<tr><td>HS</td><td>NI3</td><td>NI2a</td><td>NI2a</td></tr>
<tr><td>HS</td><td>NI2a</td><td>NI3</td><td>NI2a</td></tr>
</table></td></tr>
<tr><td colspan="2">Experimental Control Specimens</td><td colspan="4">none required</td></tr>
<tr><td colspan="2">Acceptance Criteria
(torque in inch-pounds)</td><td colspan="2">Bolt Size (inches) - Thread Pitch
0.250 - 28</td><td>0.500 - 20</td></tr>
<tr><td colspan="2">Optimum self-locking run-on/run-off torque</td><td colspan="2">30</td><td>150</td></tr>
<tr><td colspan="2">Maximum self-locking run-on/run-off torque*</td><td colspan="2">75</td><td>375</td></tr>
<tr><td colspan="2">Minimum breakaway torque</td><td colspan="2">3.5</td><td>18</td></tr>
<tr><td colspan="2">Suggested range of ratio of breakloose torque to assembly torque</td><td colspan="2">1.5 to 3</td><td>1.5 to 3</td></tr>
<tr><td colspan="2">Maximum ratio of breakloose torque to assembly torque*</td><td colspan="2">4</td><td>4</td></tr>
</table>

* The values marked are “hard limits” that will determine whether or not testing continues with any given candidate DFL

Major or Unique Equipment

- Oven (air circulating furnace)
- Piezoelectric load cell (force washer)
- Torque sensor
- X-Y recorder

Data Recording and Calculations

- Report the beginning and ending lengths of each bolt, to the nearest 0.0001 inch.
- Report force, torque, and angle of turn data collected while performing the room temperature calibration procedure.
- Report the angle of turn achieved for each nut/bolt assembly subjected to elevated temperatures.
- Report condition of each nut and bolt after each assembly/disassembly cycle.
- Use the following definitions in recording and reporting data:
    - Self locking run-on torque - torque required to continue motion (installation) of nut once it has been initiated
    - Grab torque - torque required to continue motion when the nut washer face contacts the block surface, indicated by sharp spike in torque required to continue motion
    - Assembly torque (clamp torque) - torque at final assembly, after the desired angle of turn (and clamping force) has been achieved
    - Breakloose torque - torque required to initiate movement of nut away from the assembled condition (measured in the loosening direction)
    - Breakaway torque - torque required to restart motion in the loosening direction when no axial load is present
    - Self locking run-off torque - torque required to continue motion (removal) of nut once the motion has been initiated

3.17. Volatile Organic Compound Content

Application Categories - LG, HG, LS, HS, AD

Test Description

This is a procedure for determining the VOC content of a DFL as supplied, in grams per liter (g/L) excluding water and exempt solvents.

Measure the VOC content of the candidate DFL as supplied in accordance with ASTM D3960 - 92 (*Standard Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings*, approved February 28, 1992). This procedure uses the following steps:

1. Determine the percent (by weight) volatile compounds in a sample in accordance with ASTM D2369 - 92 (*Standard Test Method for Volatile Content of Coatings*, approved May 15, 1992).
2. Measure the water content (in weight percent) of a sample in accordance with ASTM D3792 - 91 (*Standard Test Method for Water Content of Water-Reducible Paints by Direct Injection Into a Gas Chromatograph*, approved May 15, 1991) or ASTM D4017 - 90 (*Standard Test Method for Water in Paints and Paint Materials by Karl Fischer Method*, approved May 25, 1990); note that the procedure is not necessary if the water content is known to be zero.
3. Determine the density of the DFL as supplied (in its fluid form) in accordance with ASTM D1475 - 90 (*Standard Test Method for Density of Paint, Varnish, Lacquer, and Related Products*, approved October 26, 1990).
4. Measure the weight percent of exempt solvents in accordance with ASTM D4457 - 85 (*Standard Test Method for Determination of Dichloromethane and 1,1,1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph*, approved July 16, 1985, reapproved July 1991); note that this procedure is not necessary if the exempt solvent content is known to be zero.
5. Calculate the VOC content of the DFL as supplied (fluid form) as detailed in ASTM D3960 - 92.

Rationale

Measuring the VOC content of the DFL is necessary to determine what personal protective equipment and engineering controls will be required to safeguard worker health and safety during application of the DFL. Choosing DFLs at least partly on the basis of low VOC content helps ensure that the environmental problem of lead is not replaced with excessive VOC content. The VOC content used as the acceptance criteria below was chosen by the participants in the JG-PP/ PEWG DFL Project based on VOC limits imposed on the individual OEMs by federal, state, and local regulations and by corporate policies.

Note: The Army Fuels and Lubricants Technology Team (U.S. Army Mobility Technology Center), as the preparing activity for MIL-L-46010E (*Lubricant, Solid Film, Heat Cured, Corrosion Inhibiting*; issued April 11, 1997) and MIL-L-46147B (*Lubricant, Solid Film, Air Cured (Corrosion Inhibiting)*; issued December 2, 1994), has the authority for setting the acceptable VOC content limits for solid (dry) film lubricants for dry film lubricants meeting those specifications. In accordance with Department of

Defense pollution prevention policy, the "Fuels and Lubes" team has limited VOC content (less water and exempt solvents) of solid film lubricants (also known as DFLs) to 250 g/L, to meet current or expected limits imposed by the South Coast Air Quality Management District (SCAQMD, located in southern California). This decision was made at least in part because DFLs that had low VOC content and met the performance requirements of MIL-L-46010E and MIL-L-46147B were available.

The OEMs participating in the joint JG-PP/PEWG effort to eliminate the use of lead-containing DFLs are not subject to the DoD policy limiting DFL VOC content to no more than 250 g/L, and none of their manufacturing facilities are located in the SCAQMD. In addition, the performance requirements contained in this JTP are in some ways more stringent than those specified in MIL-L-46010E or MIL-L-46147B. The participants believe that their goal of eliminating lead from the DFLs used while maintaining performance characteristics should not be compromised by imposing an extremely low VOC content ceiling during this effort.

According to the December 13, 1996, amendment to SCAQMD Rule 1124 (*Aerospace Assembly and Component Manufacturing Operations*, amended December 13, 1996) solid film lubricants used for fastener installation must not exceed 880 g/L VOC content and lubricative coatings applied to components other than fasteners must not exceed 880 g/L VOC content. According to the same rule, the VOC content of solid film lubricant types of lubricative coatings applied to fasteners must not exceed 800 g/L at present, and will be limited to 250 g/L VOC content beginning in January 1998.

The Environmental Protection Agency's draft Control Technique Guidelines document for controlling volatile organic compound (VOC) emissions from aerospace manufacturing and rework facilities also specifies that solid film lubricants applied in an uncontrolled manner (not confined to a spray booth, etc.) should contain no more than 880 g/L VOC. The Control Technique Guidelines (CTG) are intended to assist states in analyzing and determining reasonably available control technology (RACT) for stationary sources of VOC emissions located within ozone national ambient air quality standard nonattainment areas, and are not federal regulations. Individual states may impose more stringent rules than those suggested in the CTG. Note that at the time of writing this JTP, the National Emission Standards for Hazardous Air Pollutants (Air Toxics Standard) For Aerospace Manufacturing And Rework Facilities does not specify any limits on VOC content of solid film lubricants (DFLs).

Where the National Emission Standards for Hazardous Air Pollutants (NESHAP) specifies VOC content, it states that VOC content should be determined by Method 24. The procedure prescribed by ASTM D3960 - 92 parallels the intent of Method 24 for measuring the content of nonaqueous, nonexempt volatile matter (solvents).

Test Methodology

Parameters	As required by the ASTM methods used
Number and Type of Specimens per Candidate DFL	2 samples as required by the ASTM methods used
Experimental Control Specimens	None required
Acceptance Criteria	VOC content no greater than 500 g/L for DFLs supplied as bulk liquid and VOC content no greater than 880 g/L for DFLs supplied in aerosol cans

Major or Unique Equipment

- Oven (forced draft oven or air circulating furnace) (ASTM D2369 - 92)
- Desiccator (ASTM D2369 - 92)
- Aluminum foil sample dishes (ASTM D2369 - 92)
- Gas chromatograph with appropriate columns, charging devices, and recorder (ASTM D3792 - 91 and ASTM D4457 - 85)
- Karl Fischer Apparatus (ASTM D4017 - 90)
- Constant temperature bath (ASTM D1475 - 90)
- Thermometers (ASTM D1475 - 90)
- Pycnometer or weight-per-gallon cup (ASTM D1475 - 90)
- Laboratory balance (ASTM D1475 - 90)

Data Recording and Calculations

Report all intermediate data, and the VOC content in g/L.

Data Analysis (conforming to ASTM D3960 - 92)

VOC content (VOC) in the DFL as a liquid is:

$$VOC = \frac{\left[(W_v - W_w - W_{ex}) * D_c\right]}{\left[100\% - W_w * \left(D_c / D_w\right) - W_{ex} * \left(D_c / D_{ex}\right)\right]}$$

where

VOC =	VOC content in g/L of coating less water and exempt solvents
W_v =	Weight percent of total volatiles (see ASTM D2369 - 92)
W_w =	Weight percent of water (see ASTM D3792 - 91 or ASTM D4017 - 90; $W_w = 0$ if the DFL is known to contain no water)
W_{ex} =	Weight percent of exempt solvents (see ASTM D4457 - 85; $W_{ex} = 0$ if the DFL is known to contain no exempt solvents)
D_c =	Density of DFL in fluid form at 25°C in g/L (see ASTM D1475 - 90)
D_w =	Density of water at 25°C in g/L ($0.997*10^3$)
D_{ex} =	Density of exempt solvents at 25°C in g/L (see ASTM D1475 - 90)

3.18 Humidity Resistance

Application Categories – LG, HG

Test Description

This test allows for the evaluation of the re-hydration potential of a DFL coating applied to metallic alloys and exposed to high humidity, to determine whether the DFL can be easily removed from the surface after re-hydration occurs.

Apply DFL to 3" x 6" x 0.020" thick test panels as recommended by the manufacturer, to a thickness of 0.9 to 1.1 mils. Verify the film thickness by measurement at three separate locations on each panel. Visually examine the panels under normal working light and 3x magnification, to verify that the applied DFL is a smooth film of uniform color, with no cracks, sags, runs, scratches, pinholes, blisters, nodules, or chipping.

Evaluate the potential of a candidate DFL to re-hydrate by placing coated test panels into a humidity cabinet operating at 120°C and 100% relative humidity . Panels are assessed at an interval of 168 hours (weekly) for a minimum total of 2016 hours (12 weeks) by wiping the surface once with moistened filter paper. Significant removal of the coating indicates potential failure of the DFL by insufficient material remaining on the part. In addition to visually inspecting each panel to verify that no base metal is exposed, use a flat micrometer to measure each panel in six locations to 0.0001".

Rationale

Exposure of a DFL to hot, humid conditions has the potential to rehydrate the binder, rendering the DFL as a "wet" coating that is susceptible to removal.

Test Methodology

Parameters	120°F ± 10°F (48°C ± 12°C) at 100% relative humidity for up to 2016 hours
Number and Type of Specimens per Candidate DFL	• For LG DFLs: 3 TI1a panels placed in humidity chamber, 3 TI1a panels for experimental control • For HG DFLs: 3 NI3 panels placed in humidity chamber, 3 NI3 panels for experimental control
Experimental Control Specimens	One (1) experimental set of control specimens consists of 3 panels not placed in the chamber but held in a desiccator over the same time period
Acceptance Criteria	Minimal or no removal of DFL coating shall be observed over the length of the testing

Major or Unique Equipment

- Humidity Chamber

Data Recording and calculations

- Report three (3) thickness measurements per coated panel.
- Report condition of test panels and observation of filter paper.
- Report six (6) thickness measurements per coated panel to 0.0001" with a flat micrometer. Report the average for each coated panel.

4. QUALITY CONTROL TESTS

The tests described below were identified by the participants in the JG-PP/PEWG effort as necessary to implementation of any acceptable candidate DFLs. These tests are not required to validate the performance of DFLs.

Table 5. Quality Control Tests

Test	JTP Section	Application Categories	Acceptance Criteria	References
Endurance Life	4.1	LG, HG	Acceptance criteria will depend on correlation with qualification tests	ASTM D2625 - 90
Load Carrying Capacity	4.2	LG, HG	Acceptance criteria will depend on correlation with qualification tests	ASTM D2625 - 90

4.1. Endurance Life

Application Categories - LG, HG

Test Description

This test measures the time to failure of a DFL under a known compressive force, and contributes to estimation of the useful life of the dry film lubricant.

Coat steel V-blocks and pin with the candidate dry film as recommended by the manufacturer, and cure according to the manufacturer's instructions. Load the V-blocks and pin into the test apparatus, and apply load as specified in ASTM D2625 - 90 (*Standard Test Method for Endurance (Wear) Life and Load-Carrying*, approved April 27, 1990), Procedure A. Record the time until failure of the DFL at a load of 1,000 pounds force.

Rationale

This test of endurance life is specified in MIL-L-46010E (*Lubricant, Solid Film, Heat Cured, Corrosion Inhibiting*; issued April 11, 1997) for Type I DFLs, and in MIL-L-46147B (*Lubricant, Solid Film, Air Cured (Corrosion Inhibiting)*; issued December 2, 1994) for Type I DFLs.

Test Methodology

Parameters	Applied compressive load of 1,000 pounds
Number of Trials	4
Acceptance Criteria	Acceptance criteria will depend on correlation with qualification tests

Major or Unique Equipment

- Falex Pin and V-Block (Vee Block) Test Machine
- Load Gage

Recording and Calculations

Report the average of four tests.

4.2. Load Carrying Capacity

Application Categories - LG, HG

Test Description

This test measures the maximum compressive force the DFL applied to a rotating pin can withstand for a short time, and aids in estimation of the acceptable load in use.

Coat steel V-blocks and the pin with the candidate dry film lubricant as recommended by the manufacturer and cure according to the manufacturer's instructions. Load the V-blocks and pin into the test apparatus and apply load as specified in ASTM D2625 - 90 (*Standard Test Method for Endurance (Wear) Life and Load-Carrying*, approved April 27, 1990), Procedure B. Record the maximum load the DFL can sustain for one (1) minute; failure is indicated by a sharp increase in steady-state torque or by breakage of the pin.

Rationale

This test of load carrying capacity is specified in MIL-L-46010E (*Lubricant, Solid Film, Heat Cured, Corrosion Inhibiting*; issued April 11, 1997) for Type I and Type III DFLs, and in MIL-L-46147B (*Lubricant, Solid Film, Air Cured (Corrosion Inhibiting)*; issued December 2, 1994).

Test Methodology

Parameters	Increasing compressive load (increase to failure)
Number of Trials	4
Acceptance Criteria	Acceptance criteria will depend on correlation with qualification tests

Major or Unique Equipment

- Falex Pin and Vee Block Test Machine
- Load Gage

Data Recording and Calculations

Report the average of four tests.

5. REFERENCE DOCUMENTS

The following documents in Table 6 were referenced in the development of the JTP.

Table 6. Reference Documents

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
AMS 2430L	Shot Peening	July 1993	All	2	All
AMS 2481F	Phosphate Treatment, Anti-chafing	September 1994	All	2	All
AMS 4037M	Aluminum Alloy Sheet and Plate 4.4Cu 1.5Mg 0.60Mn Solution Heat Treated	January 1993	All	2	All
AMS 4375J	Sheet and Plate, Magnesium Alloy 3.0Al 1.0Zn 0.20Mn, Annealed and Recrystallized	January 1991 (reaffirmed May 1995)	All	2	All
AMS 4911H	Titanium Alloy Sheet, Strip and Plate, 6Al 4V, Annealed	July 1995	All	2	All
AMS 4916F	Titanium Alloy Sheet, Strip and Plate, 8Al 1Mo 1V, Duplex Annealed	April 1990 (reaffirmed April 1994)	All	2	All
AMS 4967G	Titanium Alloy, Bars, Wire, Forgings, and Rings, 6.0Al 4.0V Annealed, Heat Treatable	October 1993	All	2	All

(Table 6 continued on next page)

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
AMS 5508E	Steel, Corrosion and Heat Resistant, Sheet, Strip, and Plate, 13Cr 2.0Ni 3.0W Annealed	January 1993	All	2	All
AMS 5536L	Nickel Alloy, Corrosion and Heat Resistant, Sheet, Strip, and Plate 47.5Ni 22Cr 1.5Co 9.0Mo 0.60W 18.5Fe, Solution Heat Treated	July 1993	All	2	All
AMS 5544G	Alloy, Corrosion and Heat Resistant, Sheet, Strip, and Plate 57Ni 19.5Cr 13.5Co 4.2Mo 3.0Ti 1.4Al 0.05Zr 0.006B Consumable Electrode or Vacuum Induction Melted, Annealed	April 1991 (reaffirmed May 1995)	All	2	All
AMS 5547F	Steel, Corrosion and Heat Resistant, Sheet and Strip 15.5Cr 4.5Ni 2.9Mo 0.10N, Solution Heat Treated	October 1992	All	2	All

(Table 6 continued on next page)

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
AMS 5604D	Steel, Corrosion Resistant, Sheet, Strip, and Plate 16.5Cr 4.0Ni 4.0Cu 0.30Cb, Solution Heat Treated, Precipitation Hardenable	May 1995	All	2	All
AMS 5608C	Cobalt Alloy, Corrosion and Heat Resistant, Sheet, Strip, and Plate 40Co 22Cr 22Ni 14.5W 0.07La, Solution Heat Treated	February 1995	All	2	All
AMS 5617G	Steel Bars and Forgings, Corrosion Resistant, 12Cr 8.5Ni 1.2Ti 2.0Cu, Vacuum Induction Plus Vacuum Consumable Electrode Melted, Solution Heat Treated, Precipitation Hardenable	April 1990 (reaffirmed April 1994)	All	2	All

(Table 6 continued on next page)

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
AMS 5662J	Nickel Alloy, Corrosion and Heat Resistant, Bars, Forgings, and Rings, 52.5Ni 19Cr 3.0Mo 5.1Cb 0.90Ti 0.50Al 18Fe, Consumable Electrode or Vacuum Induction Melted, 1775 degrees F (968 degrees C) Solution Heat Treated, Precipitation Hardenable	September 1994	All	2	All
AMS 5664D	Nickel Alloy, Corrosion and Heat Resistant, Bars, Forgings, and Rings, 52.5Ni 19Cr 5.1Cb 0.90Ti 0.50Al 18Fe, Consumable Electrode or Vacuum Induction Melted, 1950°F Solution Heat Treated, Precipitation Hardenable	July 1994	All	2	All

(Table 6 continued on next page)

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
AMS 5709F	Nickel Alloy, Corrosion and Heat Resistant, Bars and Forgings, 58Ni 19.5Cr 13.0Co 4.3Mo 3.0Ti 1.4Al 0.05Zr 0.006B, Consumable Electrode or Vacuum Induction Melted, 1975F (1079C) Solution, Stabilization, and Precipitation Heat Treated	August 1994	All	2	All
AMS 5732G	Steel, Corrosion and Heat Resistant, Bars, Wire, Forgings, Tubing, and Rings, 15Cr 25.5Ni 1.2Mo 2.1Ti 0.006B 0.30V, Consumable Electrode Melted, 1800 degrees F (980 degrees C) Solution and Precipitation Heat Treated	May 1995	All	2	All

(Table 6 continued on next page)

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
AMS 5843C	Alloy, Corrosion and Heat Resistant, Bars 19Cr 36Co 25Ni 7.0Mo 0.50Cb 2.9Ti 0.20Al 9.0Fe, Vacuum Induction Plus Vacuum Consumable Electrode Melted, Solution Heat Treated, Work Strengthened and Aged	January 1995	All	2	All
AMS 5858B	Steel, Corrosion and Heat Resistant, Sheet, Strip, and Plate, 15Cr 25.5Ni 1.2Mo 2.1Ti 0.006B 0.30V, Multiple Melted, 1800 degrees F (980 degrees C) Solution Heat Treated, Welding Grade Precipitation Hardenable	April 1992	All	2	All
AMS 6359F	Steel Sheet, Strip, and Plate, 0.80Cr 1.8Ni 0.25Mo (0.38-0.43C)	April 1993	All	2	All

(Table 6 continued on next page)

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
AS 7250	Nuts, Self-Locking, Steel, Corrosion and Heat Resistant High Strength, All Metal 1200 degrees F Use, UN Thread Form	December 1991 (supersedes AMS 7250F)	All	2	All
AS 7253	Nuts, Self-locking, Nickel Alloy, UNS N07001 180 000 psi, All Metal 1400 degrees F use, UNJ Thread Form	December 1991 (supersedes AMS 7253C)	All	2	All
AS 7460	Bolts and Screws, Titanium Alloy 6Al 4V Procurement Specification	June 1992 (supersedes AMS 7460F)	All	2	All
AS 7467	Bolts and Screws, Nickel Alloy, Corrosion and Heat Resistant Forged Head, Roll Threaded, Stress-Rupture Rated	January 1991 (supersedes AMS 7467C)	All	2	All

(Table 6 continued on next page)

Please note that some SAE Aerospace Material Specifications, designated as “AMS” plus a number, have been superseded by specifications designated as Aerospace Standard (AS).

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
AS 7471	Bolts and Screws, Nickel Alloy, Corrosion and Heat Resistant Forged Head, Heat Treated, Roll Threaded 1950 degrees F Solution Heat Treatment	January 1991 (supersedes AMS 7471G)	All	2	All
AS 7475	Bolts and Screws, Work-Strengthened Alloy, Corrosion and Heat Resistant Forged Head, Roll Threaded After Aging	January 1991 (supersedes AMS 7475B)	All	2	All
AS 7477A	Procurement Specification for Bolts and Screws, Steel, Corrosion and Heat Resistant 1800 degrees F Solution Heat Treated, Aged Before Roll Threaded	April 1993 (supersedes AMS 7477D)	All	2	All

(Table 6 continued on next page)

Please note that some SAE Aerospace Material Specifications, designated as “AMS” plus a number, have been superseded by specifications designated as Aerospace Standard (AS).

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
ASTM A108 - 90a	Standard Specification for Steel Bars, Carbon, Cold Finish, Standard Quality	September 1990 (approved July 27, 1990)	All	2	All
ASTM A167 - 92b	Standard Specification for Stainless and Heat-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip	October 1992 (approved August 15, 1992)	All	2	All
ASTM B117 - 94	Standard Practice for Operating Salt Spray (Fog) Testing Apparatus	April 1994 (approved February 15, 1994)	Salt Spray (Fog) Corrosion Resistance	3.10	All
			Sulfurous Acid Spray Resistance	3.13	All
ASTM B209 - 92a	Standard Specification for Aluminum and Aluminum-Alloy Sheet and Plate	July 1992 (approved May 15, 1992)	All	2	All

(Table 6 continued on next page)

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
ASTM B244 - 79	Standard Method for Measurement of Thickness of Anodic Coatings on Aluminum and of Other Nonconductive Coatings on Nonmagnetic Basis Metals with Eddy-Current Instruments	April 1979 (approved February 12, 1979)	Cured Film Thickness Uniformity	3.3	All
ASTM B499 - 88	Standard Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals	November 1988 (approved August 26, 1988)	Cured Film Thickness Uniformity	3.3	All
ASTM D1141 - 90	Standard Specification for Substitute Ocean Water	March 1990 (approved February 2, 1990, reapproved 1992)	Fluid Resistance	3.7	All
			Sulfurous Acid Salt Spray	3.13	All

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Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
ASTM D1186 - 87	Standard Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base	July 1987 (approved May 29, 1987)	Cured Film Thickness Uniformity	3.3	All
ASTM D1193 - 91	Standard Specification for Reagent Water	November 1991 (approved September 15, 1991)	Fluid Resistance	3.7	All
ASTM D1400 - 87	Standard Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base	July 1987 (approved May 29, 1987)	Cured Film Thickness Uniformity	3.3	All
ASTM D1475 - 90	Standard Test Method for Density of Paint, Varnish, Lacquer, and Related Products	December 1990 (approved October 26, 1990)	Volatile Organic Compound Content	3.17	All

(Table 6 continued on next page)

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
ASTM D1654 - 92	Standard Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments	December 1992 (approved October 15, 1992)	Salt Spray (Fog) Corrosion Resistance	3.10	All
ASTM D1730 - 67	Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting	November 1967 (approved September 8, 1967, reapproved 1993)	All	2	Type C, Method 1 and Method 2
ASTM D2369 - 92	Standard Test Method for Volatile Content of Coatings	July 1992 (approved May 15, 1992)	Volatile Organic Compound Content	3.17	All
ASTM D2510 - 83	Standard Test Method for Adhesion of Solid Film Lubricants	August 1983 (approved March 25, 1983)	Thermal Shock Stability	3.14	Procedure B (Sections 1 to 8, 9.3 to 9.8)
			Dry Tape Adhesion	3.4	Procedure A
			Fluid Resistance	3.7	Procedure C (Sections 1 to 8, 9.3 to 9.8, 11)

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Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
ASTM D2511 - 83	Standard Test Method for Thermal Shock Sensitivity of Solid Film Lubricants	August 1983 (approved March 25, 1983)	Thermal Shock Stability	3.14	Procedure B (Sections 1 to 8, 9.3 to 9.8)
ASTM D2625 - 90	Standard Test Method for Endurance (Wear) Life and Load-Carrying Capacity of Solid Film Lubricants (Falex Pin and Vee Method)	June 1990 (approved April 27, 1990)	Endurance Life	4.1	Procedure A (Sections 1 to 10, 12)
			Load Carrying Capacity	4.2	Procedure B (Sections 1 to 9, 11, 12)
ASTM D2649 - 83	Standard Test Method for Corrosion Characteristics of Solid Film Lubricants	August 1983 (approved March 25, 1983)	Aluminum Corrosion Resistance	3.1	All
ASTM D3335 - 85a	Standard Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy	January 1986 (approved July 16, 1985, and November 29, 1985, reapproved Jun 1991)	Lead and Cadmium Content	3.8	All

(Table 6 continued on next page)

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
ASTM D3718 - 85a	Standard Test Method for Low Concentrations of Chromium in Paint by Atomic Absorption Spectroscopy	January 1986 (approved May 31, 1985, and November 29, 1985, reapproved July 1991)	Chromium Content	3.2	All
ASTM D3792 - 91	Standard Test Method for Water Content of Water-Reducible Paints by Direct Injection Into a Gas Chromatograph	July 1991 (approved May 15, 1991)	Volatile Organic Compound Content	3.17	All
ASTM D3960 - 92	Standard Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings	April 1992 (approved February 28, 1992)	Volatile Organic Compound Content	3.17	All
ASTM D4457 - 85	Standard Test Method for Determination of Dichloromethane and 1,1,1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph	October 1985 (approved July 16, 1985, reapproved July 1991)	Volatile Organic Compound Content	3.17	All

(Table 6 continued on next page)

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
ASTM E376 - 89	Standard Practice for Measuring Coating Thickness by Magnetic-Field or Eddy-Current (Electromagnetic) Test Methods	December 1989 (approved October 27, 1989)	Cured Film Thickness Uniformity	3.3	All
ASTM F945 - 85	Standard Test Method for Stress-Corrosion of Titanium Alloys by Aircraft Engine Cleaning Materials	September 1985 (approved July 26, 1985, reapproved 1993)	Stress Corrosion	3.12	All
ASTM G115 - 93	Standard Guide for Measuring and Reporting Friction Coefficients	May 1993 (approved March 15, 1993)	Reciprocating Sliding Wear	3.9	All
MIL-A-8243D	Anti-Icing and Deicing-Defrosting Fluids	December 21, 1989	Fluid Resistance	3.7	All
MIL-H-87257	Low Temperature Fire Resistant Aircraft and Missile Hydraulic Fluid, Synthetic Hydrocarbon Base	March 2, 1992 (interim amendment 1 January 11, 1995)	Fluid Resistance	3.7	All
MIL-L-23699E	Synthetic Base Aircraft Turbine Engine Lubricating Oil	August 25, 1994	Fluid Resistance	3.7	All

(Table 6 continued on next page)

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
MIL-L-46010E	Lubricant, Solid Film, Heat Cured, Corrosion Inhibiting	April 11, 1997	Aluminum Corrosion Resistance	3.1	3.4.6, 4.5.2.1, 4.6
			Fluid Resistance	3.7	3.4.2, 4.6
			Salt Spray (Fog) Corrosion Resistance	3.10	3.4.8, 4.5.2.2, 4.6 (Table I)
			Sulfurous Acid Salt Spray	3.13	3.4.7, 4.5.2.2, 4.6 (Table I)
			Thermal Shock Stability	3.14	3.4.3, 4.6, 3.4.1
			Endurance Life	4.1	3.4.4, 4.6
			Load Carrying Capacity	4.2	3.4.5, 4.6
MIL-L-46147B	Lubricant, Solid Film, Air Cured (Corrosion Inhibiting)	December 2, 1994	Fluid Resistance	3.7	3.9, 3.4, 4.6
			Salt Spray (Fog) Corrosion Resistance	3.10	3.7, 4.6
			Thermal Shock Stability	3.14	3.4, 3.8, 4.6 (Table II)
			Endurance Life	4.1	3.5, 4.6
			Load Carrying Capacity	4.2	3.6, 4.6

(Table 6 continued on next page)

Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
MIL-P-23377G	Primer Coatings: Epoxy, High-Solids	September 30, 1994	Chromium Content	3.2	1.2.2, 3.4.1.2
MIL-P-85582B	Primer Coatings: Epoxy, Waterborne	May 23, 1994 (interim amendment 1 August 31, 1994)	Chromium Content	3.2	1.2.2, 3.4.2.3
MIL-T-5624R	Aviation Turbine Fuel, Grades JP-4, JP-5, and JP-5/JP-8 ST	March 3, 1995	Fluid Resistance	3.7	All
MIL-T-83133D	Aviation Turbine Fuel, Kerosene Types, NATO F-34 (JP-8) and NATO F-35	January 29, 1992	Fluid Resistance	3.7	All
P-D-680B	Dry Cleaning and Degreasing Solvent	October 29, 1992 (interim amendment 2 May 16, 1995)	Solvent Rub	3.11	All
QQ-P-35C	Passivation Treatments for Corrosion-Resistant Steel	October 28, 1988	All	2	All
TT-P-664D	Primer Coating, Alkyd, Corrosion-Inhibiting, Lead and Chromate Free, VOC-Compliant	September 1, 1988	Lead and Cadmium Content	3.8	Table III

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Table 6. Reference Documents (continued)

Reference Document	Title	Date	JTP Test	JTP Section	Applicable Section(s) of Reference Document
VV-D-1078B	Silicone Base (Dimethyl Polysiloxane) Damping Fluid	November 5, 1991	Fluid Resistance	3.7	All